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Effect of Porosity and Particle Size on Microwave Heating of Copper

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Abstract:

The present study investigates the effect of varying particle size and porosity on the heating behavior of a metallic particulate compact in a 2.45GHz multimode microwave furnace. Experiments on copper suggest that unlike monolithic (bulk) materials, metallic materials do couple with microwaves when they are in particulate form. The powder compacts having higher porosity and smaller particle sizes interact more effectively with microwaves and are heated more rapidly. A dynamic electromagnetic-thermal model was developed to simulate the temporal temperature distribution using a 2-D finite difference time domain (FDTD) approach. The model predicts the variation in temperature with time during heating of copper powder compacts. The simulated heating profiles correlate well with those observed from experiments.

Keywords: *Finite Difference Modelling (FDM); Microwave heating; Sintering; Numerical Modeling, Heat Transfer*

Introduction

Microwaves are primarily coherent, polarized electromagnetic waves which lie between the radio-waves and visible light in the electromagnetic spectrum. Their frequency lies between 0.3 to 300 GHz and wavelength varies from 1 mm to 1 m range. One of the key applications of microwaves is to heat materials. For such applications, however, only few frequencies are allowed, namely 915 MHz, 2.45 GHz, 5.8 GHz and 28 GHz [1]. Out of these 2.45 GHz multimode microwaves are most commonly used. For engineering applications, microwaves are known to interact strongly with a range of ceramics – both in bulk as well as particulate form - and heat them to high temperatures (up to 1800°C) [2]. Unlike conventional heating, wherein the heat-transfer is limited by radiative and conductive mode, microwaves directly couple with ceramics result in rapid and uniform heating. Microwave heating of ceramic and other dielectrically lossy materials have been widely investigated and the mechanism of microwave-material interaction is well documented [2-4]. Microwave penetrates and propagates through dielectric material. Thus, in effect, the material per se becomes the source of heat. Consequently, at times, the flow of heat is the reverse of that observed in conventional heating.

Unlike ceramics, bulk or monolithic metallic materials are known to reflect

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microwaves at room temperature. Being good conductors, no internal electrical field is induced in metallic materials. The induced electrical charges remain restricted at the surface of a bulk metallic sample. This causes the eddy currents and subsequently resistive losses. Roy et al. [5] were the first to recognize that this can be exploited to heat metals too to high temperatures provided they are in particulate form. This opened the possibility of consolidating the particulate metal compacts to full density through sintering using microwaves. Subsequently, several researchers [6-19] have demonstrated that a range of particulate metals and alloys can be heated using microwaves and can result an overall 60 to 90% reduction in the processing time. Besides cost advantage, a reduced processing time restricts microstructural coarsening that occurs concomitantly with densification during sintering. Hence, as compared to compacts processed in a conventional (radiatively-heated) furnace, microwave sintered compacts have more homogenous and refined microstructure which leads to an improved mechanical properties [8-19].

While the efficacy of microwaves to sinter metals is now well recognized, a systematic evaluation of factors influencing the microwave interaction with the particulate compact and its influence on the heating rate is as yet lacking. Recently, Saitou [10] systematically investigated microwave heating of several metal powder compacts and reported that the sintering mechanism per se remain the same both in conventional as well as microwave heating. In recent years, using single-mode microwaves where the electric (E) and magnetic (H) fields were separated; researchers [20-23] have demonstrated experimentally that the effect of microwave frequency, sample conductivity, and green density on heating efficiency of metallic compacts. However, from an industrial materials processing standpoint, single-mode microwaves have limited application. Mishra et al. [24] modeled microwave heating in metallic systems by solving the Maxwell's equation and using a two-dimensional finite difference time domain (FDTD) technique. However, their model ignored the influence of the initial compact porosity and was not validated experimentally.

From the preceding discussion, it is evident that while microwave heating of ceramics is well-understood and many models have been attempted, there is still an uncertainty about the exact mechanism and mode of microwave heating in case of particulate metals. This study therefore sets out to systematically investigate - both experimentally as well as through a thermal-electromagnetic model - the effect of initial powder size and the as-pressed porosity on the heating response of a copper compact in a multimode (2.45 GHz) microwave furnace.

2. Modeling approach

Modeling of microwave heating involves solving Maxwell equations of electromagnetism simultaneously with the heat transfer equation [25]. The detailed approach which has been adapted in this study to calculate the electro-magnetic absorption (P_{EM}) by metal powder compact has been discussed elsewhere [24].

In the present study, the calculated power absorbed is correlated with the temperature rise in the compact. The latter requires solving the heat transfer equation for the boundary conditions defined by the dimensions of the sample and its physical, thermal and dielectric properties. The dependency of these parameters on temperature necessitates the need for a dynamic simulation. Figure 1 shows the modeling approach adopted in this study through a schematic flow-chart.

Microwaves interaction with metals is restricted to its surface only. This depth of penetration in metals, also known as skin-depth (δ), is defined as the distance into the material at which the incident power drops to 1/e (36.8%) of the surface value. The skin depth is mathematically expressed as follows [26]:

$$\delta = \frac{1}{\sqrt{\pi f \mu \sigma}} = 0.029\sqrt{\rho \lambda_o} \tag{1}$$

where, f is normal microwave frequency, μ is magnetic permeability, σ is electrical conductivity, ρ is electrical resistivity, λ_o is incident wavelength (12.24 cm for 2.45 GHz waves). The skin-depth in metals typically varies between 0.1 to 10 μm. From Equation 1, it is evident that metals with higher electrical conductivity have lower skin depths. For metals, as the resistivity increases with increase in temperature, the skin depth too increases. In general, the skin depth is relatively small in metals; however, most metallic powders typically have equivalent dimensions. In metal powders, the surface area and thereby the 'effective skin' (portion of metal powder that couples with microwaves) is high enough to contribute to its heating. Thus, it is likely that some of the submicron and nano-sized metal powders undergo volumetric heating when subjected to microwaves. The skin depth of the metal plays an important role in governing the power loss during the microwave-metal interaction which leads to its heating [25]. The tangential component of the magnetic field, H_t, of microwaves induces an electrical field, E, at the metal powder surface. The induced electrical field generates surface current which causes the resistive Joule heating in metal powders. Assuming spherical metal powder of radius, r_p, the electro-magnetic power density (P_{EM}) can be expressed as [25]:

$$P_{EM} = \frac{3P}{r_p} = \frac{6R_s|E_o|^2}{\eta_o^2 \cdot r_p} \tag{2}$$

where, R_s is surface resistivity ($R_s = 1/\sigma\delta$, σ is electrical conductivity and δ is skin depth); E_o is electric field amplitude at surface; and η_o is impedance of free space (377 Ω). In our approach E_o is considered as a tunning parameter to fit the experimental data. In our earlier work [24] the effect of porosity and particle size has not been considered whereas in this model it has been considered.

2.2. Assumptions

To make the problem tractable and simplistic for modeling, following assumptions are made:

- Since the particle size is much smaller than the wavelength of the microwave radiation, the field across the particle is assumed to be spatially uniform and periodic in time.
- Powder coarsening is not assumed during heating.
- Monosized powders are assumed so that the calculated EM-power density generated in a powder will be same when integrated over bulk aggregate.
- All metal powders within a compact are assumed to heat-up at the same rate so no heat conduction between particles occurs.
- A heat-factor is considered to account for the presence of susceptor.
- Effect of oxide layer formation is not considered in the present model.

As shown schematically through Fig. 1, E_o which is the electric field amplitude at surface are in turn fed as input parameter into the thermal model.

2.3. Thermal Model for Predicting Temperature Rise

Thermal Model for Cylindrical Geometry Compact has been defined as following:

$$\rho C_P \frac{\partial T}{\partial t} = \frac{1}{r}\frac{\partial}{\partial r}\left(Kr\frac{\partial T}{\partial r}\right) + \frac{\partial}{\partial z}\left(K\frac{\partial T}{\partial z}\right) + P_{EM} \tag{3}$$

where

ρ : Density of the material, which is time dependent and increases as densification increases.
K: Thermal conductivity of the material (temperature dependent)
C_P : Specific heat of the material (temperature dependent)
P_{EM} : Power density due to microwave effect (temperature dependent)

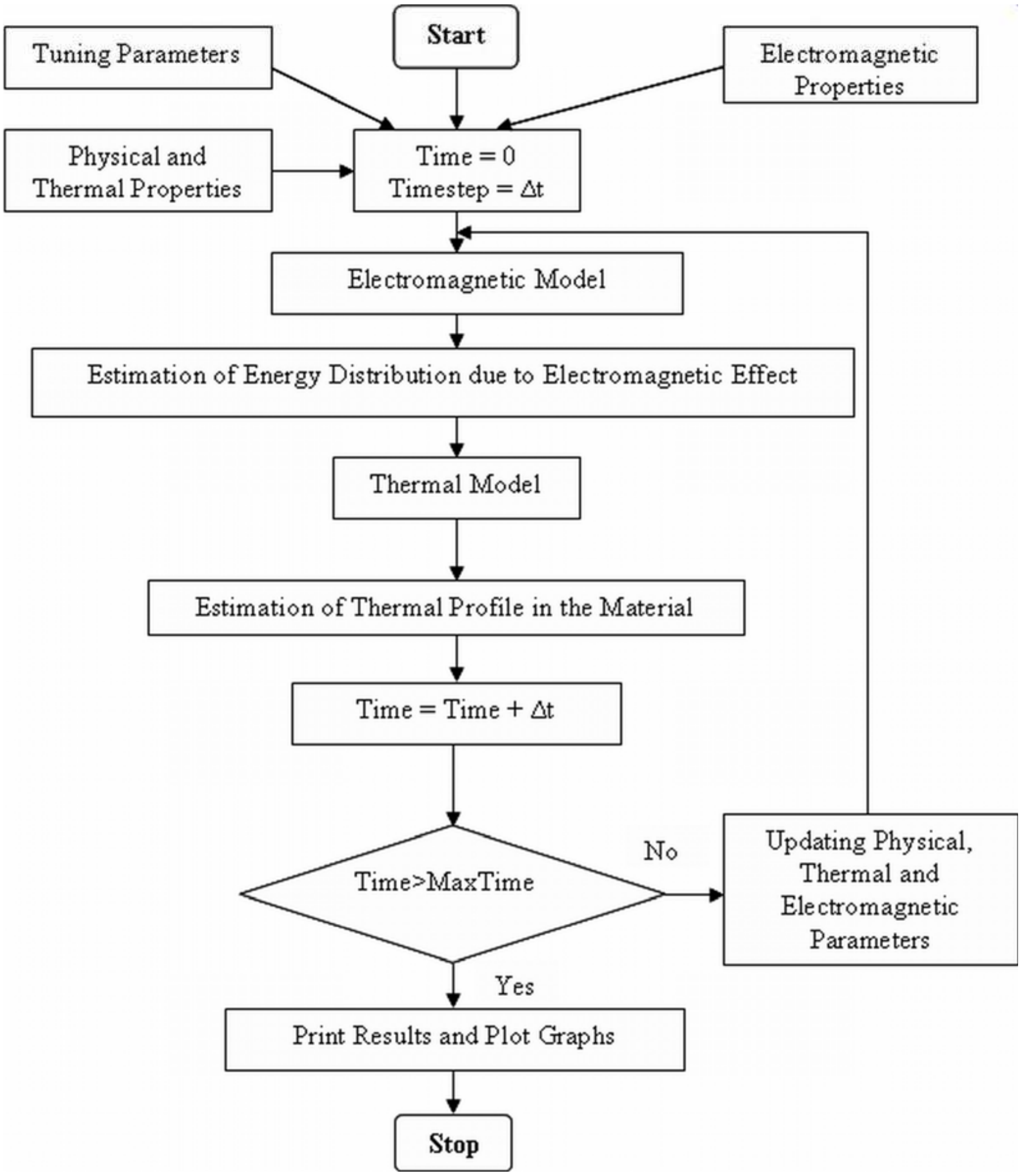

Fig. 1. Flow chart of the dynamic model proposed for simulating the thermal profile in the present study.

Typically, in the initial stage of heating, the metal powder compacts require susceptor-assisted heating [27]. The susceptors usually couple very well with the microwaves and are used for initially raising the temperature of the compact. Beyond a critical temperature, the heating occurs primarily due to microwave-metal powder surface interaction. Besides, assisting heating in the initial stage, the main role of susceptor is to prevent heat loss from the surface of the powder compact to the surrounding through radiation. In the present modeling scheme, the contribution of SiC as susceptor for compact heating was also included. Hence, the boundary condition at the surfaces of the compact can be formulated as following:

$$-K\frac{\partial T}{\partial r} = h(T - T_a) + K_{SUC}(T - T_{SUC}) + \sigma\varepsilon F(T^4 - T_{encl}{}^4) \tag{4}$$

where

H: Heat Transfer Coefficient for convective heat transfer to surrounding

T_{SUC} : Temperature of the SiC suspector

T_{encl} : Temperature of the enclosure in which compact is put

T_a : Ambiant Air temperature

K_{SUC} : Susceptor constant

σ : Stefens-Boltzman's constant

ε : Emissivity

F : View Factor

When no thermal gradient inside the compact exists, above equations may be written as following:

$$-\rho C_P\left(\frac{V}{A}\right)\frac{\partial T}{\partial t} = h(T - T_a) + K_{SUC}(T^4 - T^4{}_{SUC}) + \sigma\varepsilon F(T^4 - T_{encl}{}^4) - P_{EM}\left(\frac{V}{A}\right) \tag{5}$$

where

$$\frac{V}{A} = \frac{\pi R^2 H}{\left(2\pi RH + 2\pi R^2\right)}$$

h is convective heat transfer coefficient of incoming gas,

A/V is surface area to volume ratio for compact,

σ is Stefan Boltzman constant,

ε is effective emissivity of metal powder, and

T_a is surrounding temperature.

Above equation may be discretisized with respected to time in following manner:

$$\frac{T_{n+1} - T_n}{\Delta t} = \frac{\partial T}{\partial t} \tag{6}$$

In Equation 6, Δt should not be too small as it increases the number of iterations, hence, the round-off error is also increased. However, too large a Δt should also be avoided as it causes an increase in the truncation error per step. Therefore, a suitable value of Δt is chosen which satisfies stability criterion given below:

$$0.05 \le \Delta t \cdot K \le 0.1 \tag{7}$$

where, K is upper bound of following expression:

$$\left| d\left\{\rho C_p\left(\frac{A}{V}(h(T - T_a) + K_{SUC}(T - T_{SUC}) + \sigma\varepsilon F(T^4 - T^4_{encl}) - P_{EM}\right)\right\}\frac{1}{dT}\right|$$

3. Experimental setup to validate model

The as-received gas-atomized copper powders (supplier: American Chemet Corp. USA) of different particle size were characterized for their size, size distribution and morphology. Tab. I summarizes the characteristics of the as-received powders used for this study. Fig. 2 shows the scanning electron micrographs of the powders in as-received condition. The particle size analysis of the as-received powders was performed using a Malvern Instruments particle size analyzer which works on the principle of laser diffraction.

Tab. I. Characteristics of the as-received Cu powders used for the present study.

Cu Powder Grade	A	B	C	D	E
Purity, %	~99	>99.5	>99.2	>99.2	>98
Particle size, µm					
D_{10}	2	6	8	26	231
D_{50}	6	12	18	63	383
D_{90}	36	20	33	118	560
Specific surface area,	2.5	1.4	0.5	0.3	0.3

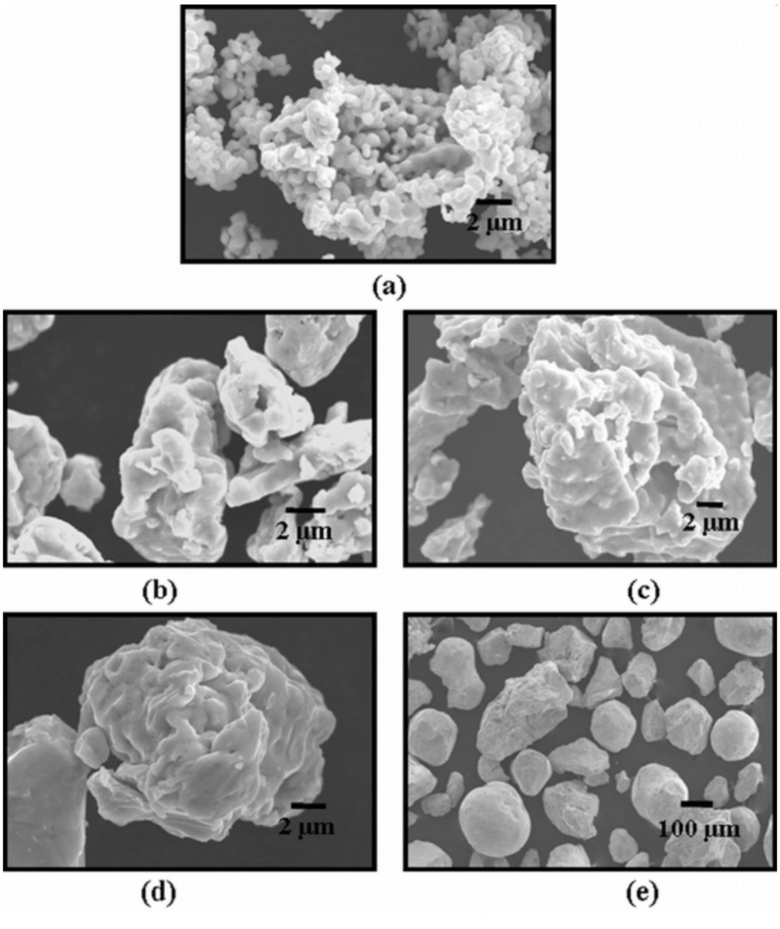

Fig. 2. SEM micrograph Cu powders used in the present study and having average particle size of (a) 6 µm, (b) 12 µm, (c) 18 µm, (d) 63 µm and (e) 383 µm, respectively.

The surface areas of the as received powders were measured using a BET apparatus. The morphological analyses of the powders were carried out using SEM imaging. Tab. II summarizes the physical and thermal properties of the copper powder used for the present investigations, which are the input parameters in the model. The total experimental approach consists of two sub sets. First experiment is all about the effect of particle size during the heating of powder particle in a multimode microwave furnace. Second experiment includes the role of initial density/porosity during the heating of metal powders in a multimode microwave furnace. For the first experiment powders were uniaxially pressed at 400 Mpa in to compacts with green densities ranging between 60 to 83%Th. The sintering response on densification and microstructures were evaluated on cylindrical pellets (12.7 mm diameter and 5 mm average height). For the second set of experiments cylindrical pellets of the same dimensions of as-received copper powder (particle size 18 µm) were compacted using the varying compaction pressure in the range of 90 to 350 Mpa to obtain green densities ranging between 56 to 76%Th.

Tab. II. Physical and thermal properties of Cu used for simulation of the thermal profile [28,29].

Density, gcm^{-3}	8.9
Heat capacity (C_p), $Jkg^{-1}K^{-1}$	$92 \bullet 10^{-3}T + 355.9$
Electrical resistivity, µΩ-cm	6.9×10^{-3} (T-273) + 1.5
Emissivity	0.65

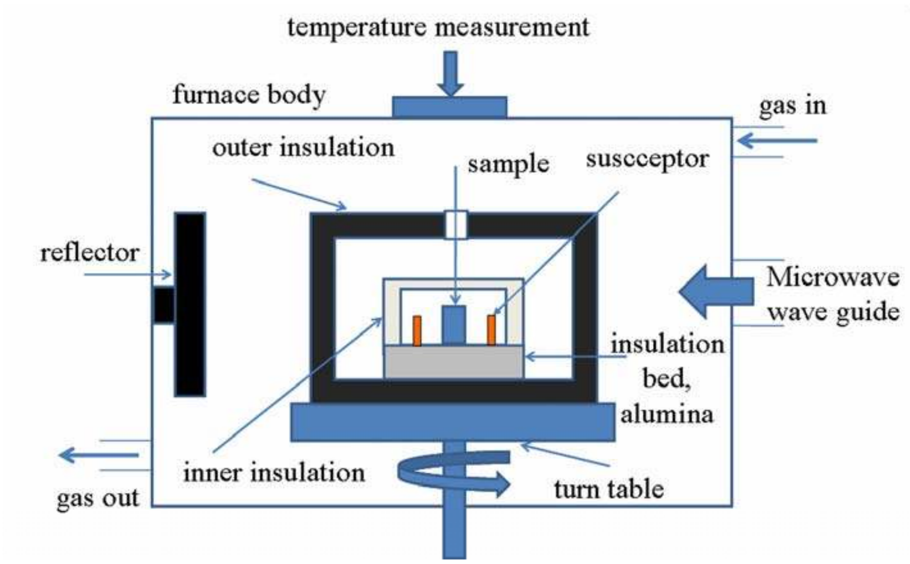

Fig. 3. Schematic diagram showing the inside of the multimode microwave furnace used in the present study.

Microwave heating of the as-pressed compacts was carried out using 2.45 GHz multimode microwaves in a 6kW capacity furnace. The experimental setup for the furnace is shown in Figure 3. A multi- layered insulation package was used to provide sufficient insulation to obtain high and uniform temperatures throughout the sample. The outer package was made up of thick ceramic fiber (aluminum silicate) sheets. A mullite tube was placed at the centre of the package, and samples were placed inside this mullite tube. The entire package was placed on a turntable to ensure uniform exposure of the sample to the microwave field. Further details about the experimental setup are described elsewhere [27]. The temperature of the sample was monitored using an infrared pyrometer (Type: RAYMA25CCF Manufacturer: Raytek Co., Santa Cruz, CA, USA). Temperature measurement through pyrometer is emissivity-based. Typically, emissivity varies with temperature. However, as very little variation in the emissivity was reported in the temperature range used in the present study, hence, the effect of variation in emissivity was ignored in the present investigation.

The sintered density was obtained by both dimensional measurements as well as Archimedes density measurement technique. The sinterability of the compact was also determined through densification parameter which is expressed as:

$$\text{Densification parameter} = \frac{(\text{sintered density - green density})}{(\text{theoretical density - green density})} \qquad \text{……… (8)}$$

4. Results and discussion

In the preceding sections, a theoretical approach has been formulated to predict the heating profile of metal powder compacts in microwave furnace. One of the first objectives was to test the efficacy of the model to predict the temperature distribution within the compact during heating. To test this, thermal profiles was simulated for Cu powder compact (particle size 18 µm) having radius 6.5 mm and 6.5 cm respectively (Fig. 4).

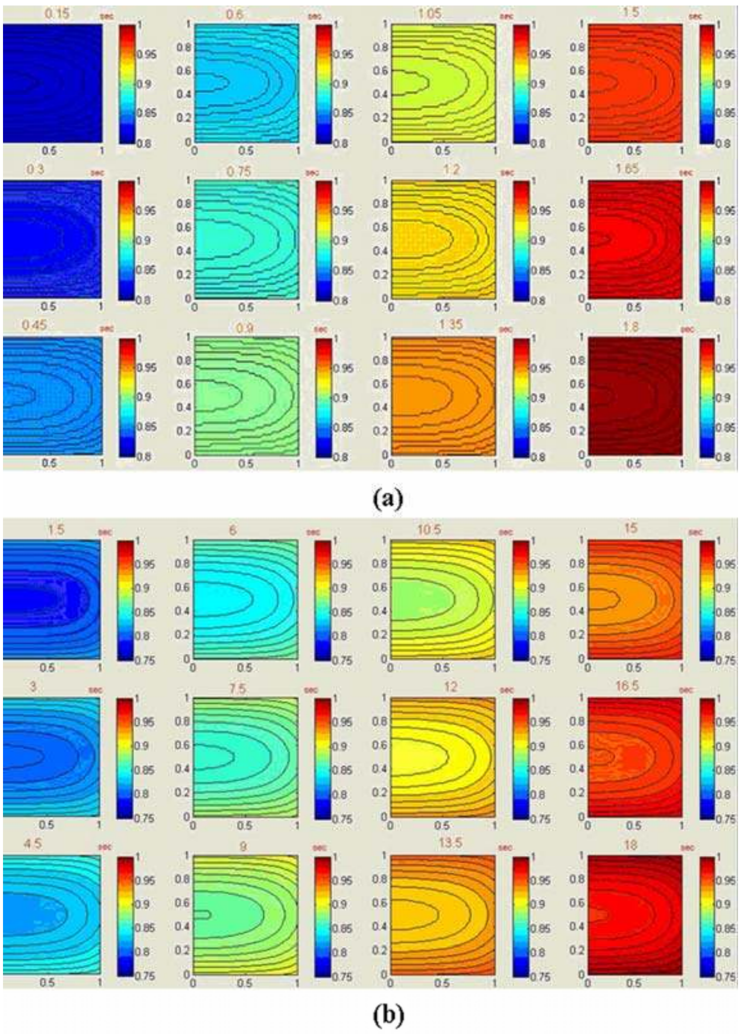

Fig. 4. Simulated isothermal contours as a function of time in cylindrical Cu compacts(Avg. particle size 18 µm) having dimensions (a) 6.5 mm radius and 5.5 mm height; and (b) 6.5 cm radius and 5.5 cm height, respectively.

As is evident from Figs. 4a and 4b, for copper powder compacts, irrespective of the compact size, the temperature remains homogenous throughout its cross-section. It is also interesting to note that for both cases, the temperature homogenization is achieved within a few seconds. Therefore the assumption that the temperature is uniform inside the compact is valid.

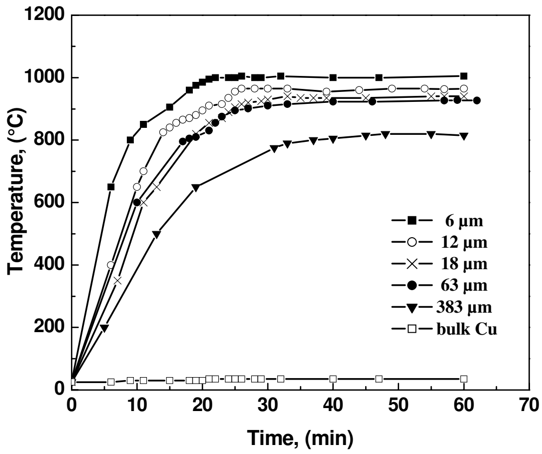

Fig. 5. Effect of varying particle size on the heating behavior of Cu compacts in a microwave furnace. For comparison the thermal profile of bulk Cu under microwave is also superimposed.

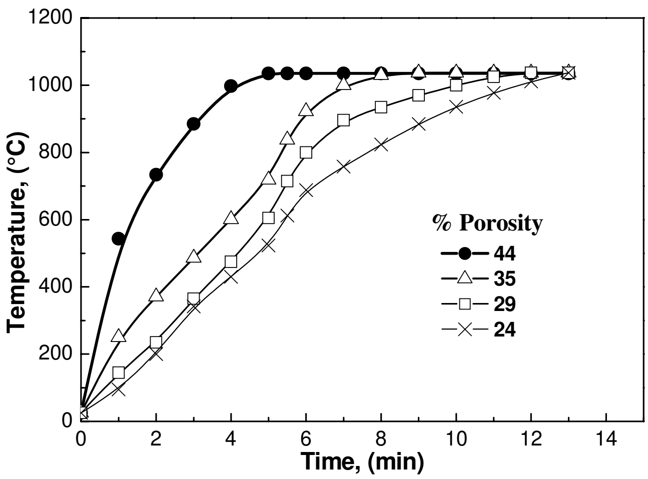

Fig. 6. Effect of varying initial porosity on the heating behavior of Cu compacts (avg. size: 18 μm) in a microwave furnace.

Figs. 5 and 6 compare the effect of the initial powder size and the as-pressed porosity on the thermal profiles of cylindrical copper compacts exposed to microwaves. For the sake of comparison, the thermal profile on bulk copper is also superimposed in Figure 5. As expected, the bulk copper does not heat up at all in microwave. In contrast, particulate compacts show strong interaction with microwaves. It is evident from the figures that the microwave heating is more effective for compacts having smaller particle size and high porosity levels. Elsewhere, Ma et al. [23] too have reported similar observations in copper powder compacts heated in single-mode microwaves.

As particle size increases the heating rate decreases and after certain time for a particular particle size; heating rate becomes constant at a particular power setting. It is well known that microwaves for electrically conducting materials such as copper do not penetrate a bulk sample beyond the skin depth. The skin depth expression in Equation 1 can be rewritten as:

$$\delta = \sqrt{\frac{2}{\mu\sigma\omega}} \tag{9}$$

where, μ is the real part of the permeability taken to be 4 $\times 10^{-7}$ T m/A for nonmagnetic materials. σ and ω are the dc electrical conductivity and angular frequency, respectively. At 2.45 GHz, the skin depth of bulk copper is about 1.3 μm. However, in case of porous compacts, the electrical conductivity correspondingly decreases. Thus, from the above equation effective skin depth also increases proportionately. The variation in the heating rates therefore can be attributed to the difference in skin depth and also the change in surface area per unit volume with the varying particle sizes. This effect is further manifested to a larger extent for coarser particle sizes on account of their higher compressibility. This is confirmed by recently report from Ma et al. [23] who have experimentally shown that the electrical conductivity of the as-pressed copper powder compacts with 22 μm powder is 10^4 times higher than those prepared using 3 μm powder.

Tab. III: Densification response of Cu powder of different particle under microwave heating.

Average Cu Powder Size (μm)	6	12	18	63	383
sintered density (% theoretical)	95.4	90.4	90.0	89.8	90.0
densification parameter	0.88	0.54	0.48	0.43	0.39

Tab. IV. Effect of compaction pressure on the densification response of Cu powder compact having average particle size of 18 μm.

Compaction Pressure (MPa)	90	210	280	350
green density (% theoretical)	56	65	71	76
sintered density (% theoretical)	78	82	87	90
densification parameter	0.50	0.49	0.55	0.58

Tab. III compares the densification response of all the sintered copper compacts of different particle sizes in microwave furnace. In case of smallest particle compacts, densification during microwave sintering is highest and the trend is similar for the rest of other particle sizes. This is also validated by comparing the densification parameters which follows similar trend as sintered density. Tab. IV compares the densification response of different initial green density copper compacts sintered in microwave furnace. Although the heating rate is faster for lower green density samples than the higher green density compacts but densification during microwave sintering is higher in case of higher green density compacts and the densification trend is also similar. This can be attributed to the difference in the initial density.

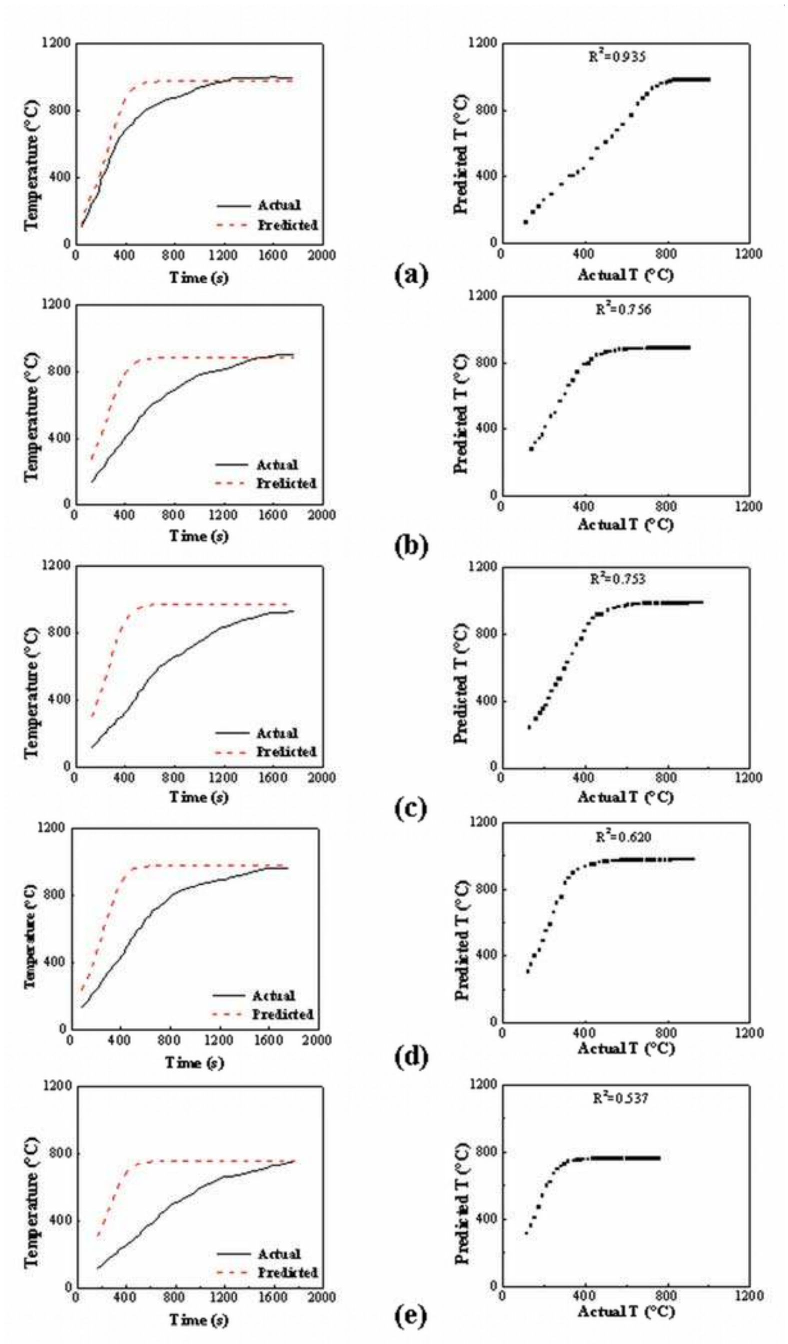

Fig. 7. Effect of varying initial particle size [(a) 6 μm, (b) 12 μm, (c) 18 μm, (d) 63 μm, (e) 383 μm] on the actual and predicted thermal profiles (*left*) and their correlation assuming a linear fit (*right*).

Figs. 7a to 7e compare the actual and predicted thermal profile of copper powder compact with increasing powder size. For all the five powder sizes, the trends in experimental and predicted heating profile are self-similar. The differences in experimental and simulated heating profiles can partly be attributed to the fact that power input was kept constant in modelling, whereas, in the actual experiment, it was changed from 0.5 kW to 1.3 kW during this experiment. An interesting observation for both experimental and simulation of thermal profile is that for a particular choice of experimental variables, the temperature rise is restricted to a certain level. This can be attributed to the fact that the heat generated in the compact (due to electromagnetic power absorbed) is balanced by the convective and radiative heat losses. The figures to the right are the corresponding regression fit for the actual and predicted temperatures for each time interval. The figures and the corresponding coefficient of correlation (R^2) values suggest that for lower particle sizes the model prediction are closer to those measured experimentally because smaller particles which size is comparable with the skin depth undergoes volumetric heating and much better microwave effect whereas powders with larger particle size undergoes only surface heating and lesser microwave induced heating effect. Smaller particle attains higher maximum temperature as compared to the larger particle.

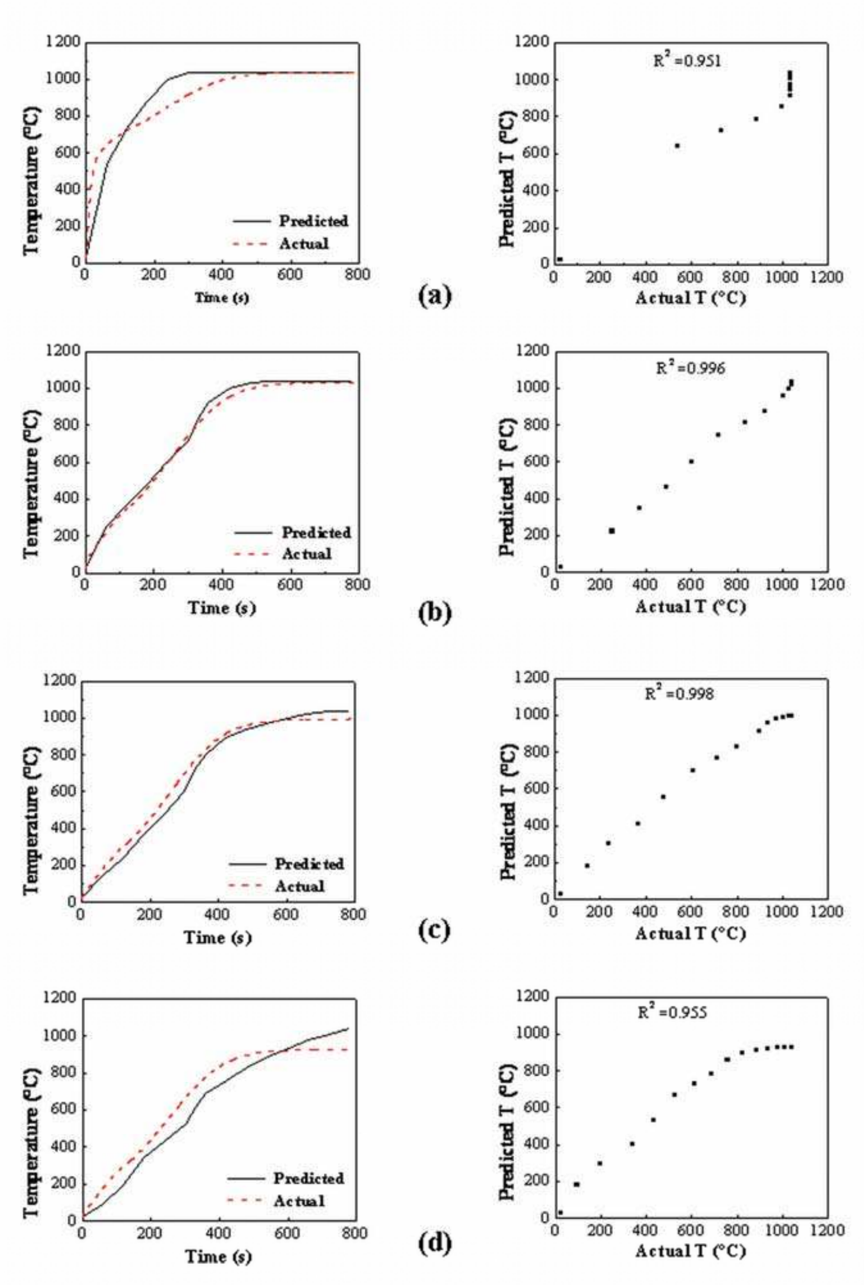

Fig. 8. Effect of varying initial porosity in a Cu compact [(a) 44%, (b) 35%, (c) 29%, (d) 24%] on the actual and predicted thermal profiles (*left*) and their correlation assuming a linear fit (*right*).

Figs. 8a to 8d compare the experimental and simulated thermal profiles of copper powder compacts (particle size: 18 μm) having initial porosity of 44%, 35%, 29% and 24%, respectively. The figures to the right are the corresponding predicted and actual temperature correlation plots. It is worth noticing that for varying porosity levels the model predictions are quite close to the experimental observations. For all the cases the correlation coefficient is greater than 0.95.

Unlike microwave heating, for conventional furnace sintering, high green density compacts are preferred so as to ensure homogenous heat transfer. The high green density requires larger compaction pressure which results in lower tool life. The relatively longer sintering time during conventional sintering is accompanied by microstructural coarsening that is detrimental to the mechanical properties. For small sized particle compacts, the problem is further compounded during conventional sintering since they have poor compressibility and are also more prone to grain coarsening. From the above discussion, it is logical to conclude that a finer powder size and higher as-pressed porosity compacts will respond more favourably when subjected to microwave heating. The present study therefore provides guidelines for effective design and optimization of process parameters to attain greater microwave heating efficacy.

5. Conclusions

This study shows that copper powder compacts strongly couple with micowaves and can be rapidly heated to high temperatures. The heating rate of the particulate compacts increases as the particle size and green density decrease. A two-dimensional FDTD approach was employed to simulate the heating response of the powder compacts that incorporated both the electromagnetic and thermal model. A special feature of this approach is that it considers the temperature-dependency on the physical and thermal properties. The predicted thermal profiles correlate well with the experimental observations.

6. Acknowledgements

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Садржај: *У овом раду проучен је утицај варирања величине честице и порозности на понашање компакта металног партикулата током загревања у 2.45 GHz мултимодалној микроталасној пећи. Експерименти вођени на бакру предлажу да за разлику од монолитичних (балк) материјала, метални материјали се куплују са микроталасима када су у форми партикулата. Компакти праха са већом порозношћу и мањим величинама ћестица реагују ефективније са микроталасима и загревају се брже. развијен је динамички електромагнетни-термички модел за симулирање темпоралне расподеле температуре коришћењем приступа дводимензионалних коначним разликама временских домена. Модел предвижа варијацију температуре са временом током загревања компакта од бакарног праха. Симулирани профили загревања су добро корелисани са експерименталним подацима.*
Кључне речи: *Моделирање коначним разликама, микроталасно загревање, синтеровање, нумеричко моделовање, пренос топлоте.*