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Extraction of effective permittivity and permeability of metallic powders in the microwave range

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Abstract
In this work, effective electric permittivity and magnetic permeability of metallic–dielectric mixtures are extracted from electromagnetic full 3D simulation data in the microwave range. The numerical method used here is the finite integration technique with periodic boundary conditions. Simulated mixtures have periodic extend in directions perpendicular to the direction of the plane wave. Thus, it is sufficient to analyze a unit element in order to extract the effective electric and magnetic properties. Using this procedure, the behavior of fine copper powders irradiated by microwaves at a frequency of 2.45 GHz is simulated. Then, the relation between particle size and the mixture’s effective properties is studied. By introducing a thin copper oxide or conductive layer it is possible to emulate the effective properties of metallic powder compacts in the early stage of sintering. Thus, this work contributes to improving the insight into the mechanisms of microwave absorption in powders of conductive materials in contrast to non-absorption in bulk metals.

Microwave sintering of ceramic powders has been a well-established method in science and industry for the last few decades [1]. Microwave heating, in contrast to conventional heating methods, allows for volumetric heating of materials, which leads to time saving and reduced energy consumption. Additionally, high heating rates in metal carbide based materials used as microwave susceptors offer a combination of microwave and conventional heating to boost the processing of less absorbing materials such as most oxides and nitrides. Rapid and controllable heating and the use of fine powders lead to a smaller grain size and a more uniform grain size distribution which improves the mechanical properties of the sintered material.

Recently microwave heating has emerged as a powerful tool for the processing of metallic powders. It was reported in 1999 by Roy et al [2] that porous metal powder compacts get heated up when subjected to microwave irradiation in either electric or magnetic field despite the well-known fact that microwaves do not penetrate bulk metals beyond skin depth and thus cannot deeply heat metals in a microwave furnace. Roy’s results show that porous metal
powder compacts are materials with both effective dielectric and effective magnetic losses, corresponding to the effective permittivity and the effective permeability of the porous metal compacts.

There are numerous experimental studies of microwave heating of metallic powders. In the recent work of Ma et al [3] microwave heating of copper powder compacts in either a magnetic or an electric field in a single mode (TE102) cavity has been studied together with measurements of magnetic and electric properties of metal compacts. The dependence of the conductivity of the sample as a function of heating time is also measured giving insight into the mechanisms of pre-sintering stage with characteristic high heating rates.

There are two important theories describing microwave absorption mechanisms in metallic powders based on experimental results. In the work by Luo et al [4] the heating rate of nickel–iron alloy powders is related to the theoretically derived formula of power absorbed by the compact. The paper of Rybakov et al [5] describes the absorption mechanism of microwaves in metallic powders with a thin oxide layer using the effective-medium approximation.

In this work, we study electric and magnetic properties of metallic powders obtained from the finite integration technique (FIT) [6] simulations. Introducing a computer model of these materials together with an extraction of effective parameters of the mixtures gives us an opportunity to have an insight into the micrometer scale microwave absorption mechanisms in metallic powders. The computer simulations are performed with the cutting-edge electromagnetic simulation software, CST Microwave Studio® 2008 (CST MWS) [7]. Further studies of the microwave heating of the metal powder compacts are performed using the finite element method (FEM) multiphysics modeling software. For this purpose we have chosen the COMSOL Multiphysics® package [8], due to its flexibility and capability to deal with coupled problems.

1. Extraction of effective properties

There are several methods for the extraction of effective material parameters for inhomogeneous mixtures. The most popular approach is the extraction from transmission and reflection characteristics of a sample. The method is based on free-space measurements of the complex permittivity and complex permeability [9, 10]. The same theory stands behind the extraction of effective properties from simulation data and is used with great success for example to describe metamaterials as homogeneous medium [11–16].

Figure 1 shows a slab of material of thickness \( d \) placed in free space. The slab is irradiated with a plane wave incident normally on the slab. Such a setup with microwave ports on the left and right sides, with a sample in between, can be considered as a two-port microwave network. The field amplitudes of the input and output waves at port 1 can be written as \( a_1 \) and \( b_1 \), respectively, and those of input and output waves at port 2 as \( a_2 \) and \( b_2 \), respectively. These parameters are either the complex field amplitudes of the electric field (\( E \)) or the magnetic field (\( H \)). The scattering parameters are collected in the scattering matrix [5]. They are used to describe the relationship between input waves \([a]\) and those of output waves \([b]\). In the case of a two-port network the output can be calculated as follows:

\[
\begin{bmatrix}
  b_1 \\
  b_2
\end{bmatrix} = \begin{bmatrix}
  S_{11} & S_{12} \\
  S_{21} & S_{22}
\end{bmatrix} \begin{bmatrix}
  a_1 \\
  a_2
\end{bmatrix},
\]

where \( S_{ij} \) are the scattering parameters with indices \( i \) denoting the destination port and \( j \) the source port. Since in our case the structure is symmetric and the plane wave is excited only at port 1, only the two scattering parameters \( S_{11} \) and \( S_{21} \) expressing the reflection and transmission characteristics, respectively, are of importance [17].
Schematic diagram of a slab, placed in free space, irradiated with a linearly polarized plane wave normally incident on the sample.

The scattering parameters $S_{11}$ and $S_{21}$ for a given frequency $f$ are related to the reflection $R$ and transmission $T$ coefficients by the following equations [10]:

$$S_{11} = \frac{R(1 - T^2)}{1 - R^2T^2},$$  
(2)

$$S_{21} = \frac{T(1 - R^2)}{1 - R^2T^2}.$$  
(3)

The reflection coefficient $R$ in free space and the slab boundary can be expressed as

$$R = \frac{Z - 1}{Z + 1}.$$  
(4)

The transmission coefficient $T$ through a homogeneous slab is

$$T = e^{-\gamma d}.$$  
(5)

In (4) and (5), $Z$ and $\gamma$ are the characteristic impedance and propagation constant of the slab, respectively. The characteristic impedance $Z$ and propagation constant $\gamma$ are related to the complex electric permittivity $\varepsilon = \varepsilon' - i\varepsilon''$ and to the complex magnetic permeability $\mu = \mu' - i\mu'':$

$$\gamma = \gamma_0\sqrt{\mu\varepsilon},$$  
(6)

$$Z = \sqrt{\frac{\mu}{\varepsilon}},$$  
(7)

where $\gamma_0 = i2\pi/\lambda_0$ is the propagation constant of free space and $\lambda_0$ the wavelength in free space. The values of $\varepsilon$ and $\mu$ are relative to free space. Rearranging (2) and (3), one obtains

$$R = K \pm \sqrt{K^2 - 1},$$  
(8)

$$T = \frac{S_{11} + S_{21} - R}{1 - (S_{11} + S_{21})R^2}.$$  
(9)

where

$$K = \frac{S_{11}^2 - S_{21}^2 + 1}{2S_{11}}.$$  
(10)
In (8) the ± sign is chosen so that |R| < 1. Rearranging (4) and inserting (7) yield

$$\sqrt{\frac{\gamma}{\varepsilon}} = \frac{1 + R}{1 - R},$$

(11)

From (6) and (11) one obtains

$$\varepsilon = \frac{\gamma}{\gamma_0} \left( \frac{1 - R}{1 + R} \right),$$

(12)

$$\mu = \frac{\gamma}{\gamma_0} \left( \frac{1 + R}{1 - R} \right).$$

(13)

By inverting (5), the propagation constant can be written as

$$\gamma = \frac{\ln(1/T)}{d}.$$  

(14)

The transmission parameter $T$ is a complex number, giving multiple values for $\gamma$. One can define $T$

$$T = |T|e^{i\phi}.$$  

(15)

Then $\gamma$ can be expressed as

$$\gamma = \frac{[\ln(1/T)]}{d} + i \left( \frac{2\pi n - \phi}{d} \right),$$

(16)

where $n$ is an integer. By inserting the value for $\gamma$ into (12) and (13) one obtains the effective complex permittivity $\varepsilon$ and complex permeability $\mu$ of the slab. The real part of $\gamma$ is unique and single valued, contrary to the imaginary part of $\gamma$ which can have multiple values. This leads to an ambiguous result, with multiple branches of the frequency dependent complex functions for $\varepsilon(f)$ and $\mu(f)$. When $d$ is large enough, these branches can lie arbitrarily close to each other, making the selection of the correct branch difficult in the case of dispersive materials. For a plane wave the phase constant $\beta$ is defined as

$$\beta = \frac{2\pi}{\lambda},$$

(17)

where $\lambda$ is the wavelength in the slab material. By comparing the expression for the phase constant $\beta$ (17) and the imaginary part of the propagation constant $\Im(\gamma)$ (16) one obtains

$$\frac{d}{\lambda} = n - \frac{\phi}{2\pi}.$$  

(18)

For $n = 0$ and $-2\pi < \phi < 0$, $d/\lambda$ is between 0 and 1. If the slab thickness $d$ is chosen such that it fulfills $d < \lambda$, then (12) and (13) will each provide unique values for $\varepsilon(f)$ and $\mu(f)$.

2. Modeling of metallic powders

The model of metallic powder consists of periodically arranged spherical particles in a cubic close packed (CCP) structure. The relative density of the CCP structure defined as a volume of non-overlapping spheres divided by the volume of the unit cell is $\rho_c = 0.74$. The CCP structure has been chosen due to its very close value of the relative density to the experimental value for copper compacts $\rho_{c(exp)} = 0.76$ [3]. The structure is excited by a plane wave (TM00 mode) with the direction of propagation along the $z$-axis, the electric field ($E$) polarized in the $x$-direction and with the magnetic field ($H$) polarized in the $y$-direction as shown in figure 2. The ports are located at the ±z limits of the mesh volume where open boundary conditions are used. A periodic boundary condition is applied at the ±x and ±y faces of the model structure.
Figure 2. Model structure as used in the simulations with CST MWS. The arrows show the polarization of the magnetic \( (H) \) and electric field \( (E) \), and the direction of propagation. Ports are at the \( \pm z \)-limits and the periodic boundary condition is applied to \( \pm x \)- and \( \pm y \)-limits. In this example the model structure contains two unit cells of copper particles arranged in the CCP structure.

An auto meshing procedure with adaptive meshing is used in the CST MWS to create the tetrahedral mesh. The number of mesh cells varies between 100k and 300k dependent on the model. The numerical problem is solved by a frequency domain solver, for frequencies from 1 to 20 GHz.

The spherical particles are made of copper. The diameter of the particles varies from 100 to 25 \( \mu m \), while the distance between the neighbouring particles is 5 nm. The obtained scattering parameters \( S_{11} \) and \( S_{21} \) are used for the extraction of effective properties.

3. Mechanisms of dielectric losses

The simulations were conducted for copper spherical particles arranged in a CCP structure containing eight unit cells, a number ascertained by convergence studies. The particles with the conductivity of bulk copper \( \sigma = 5.8 \times 10^7 \text{ S m}^{-1} \) are embedded in vacuum. The diameters of the particles used in the simulations are 3, 1, 0.5, 0.2 and 0.1 \( \mu m \). The simulations were performed for a wide frequency range from 1 to 20 GHz, and later the complex values for \( \varepsilon \) and \( \mu \) were extracted for a single frequency of 2.45 GHz. The penetration depth of bulk copper is \( \delta = 1.34 \mu m \) at the frequency of 2.45 GHz. The penetration depth of the mixture can be calculated as an inverse of the attenuation constant \( \delta = 1/\alpha \) with

\[
\alpha = \Re \left( i\omega \sqrt{\mu \varepsilon} \left( 1 - \frac{i\sigma}{\omega \varepsilon} \right) \right),
\]

where \( \omega = 2\pi f \).

The investigations were performed for two cases. In case 1, the distance between the copper particles is 5 nm of free space. The particles can be considered as separated, non-interacting absorbers. The results for case 1 are presented in table 1. In case 2, the particles are separated by a 5 nm thick oxide layer. The dielectric properties of copper oxide were taken from [18] and are \( \varepsilon' = 9.483 \) and \( \varepsilon'' = 0.9249 \). It has been reported by Ma et al [3] that the assumption of the existence of a native oxide layer is realistic to some extent. The conductivity
Table 1. Effective dielectric and magnetic properties of copper powders without the oxide layer.

<table>
<thead>
<tr>
<th>Particle diameter (µm)</th>
<th>ε′</th>
<th>ε″</th>
<th>µ′</th>
<th>µ″</th>
<th>δ (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>26.964</td>
<td>0.0006</td>
<td>0.9635</td>
<td>0.1596</td>
<td>0.046</td>
</tr>
<tr>
<td>1</td>
<td>28.117</td>
<td>0.0017</td>
<td>1.0065</td>
<td>0.0444</td>
<td>0.166</td>
</tr>
<tr>
<td>0.5</td>
<td>31.595</td>
<td>0.0019</td>
<td>1.0094</td>
<td>0.0235</td>
<td>0.297</td>
</tr>
<tr>
<td>0.2</td>
<td>32.397</td>
<td>0.0099</td>
<td>1.0048</td>
<td>0.0084</td>
<td>0.814</td>
</tr>
<tr>
<td>0.1</td>
<td>33.751</td>
<td>0.0076</td>
<td>1.0058</td>
<td>0.0078</td>
<td>0.864</td>
</tr>
</tbody>
</table>

Table 2. Effective dielectric and magnetic properties of copper powders with a thin oxide layer.

<table>
<thead>
<tr>
<th>Particle diameter (µm)</th>
<th>ε′</th>
<th>ε″</th>
<th>µ′</th>
<th>µ″</th>
<th>δ (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>27.293</td>
<td>1.1019</td>
<td>0.9631</td>
<td>0.1604</td>
<td>0.037</td>
</tr>
<tr>
<td>1</td>
<td>28.476</td>
<td>1.4143</td>
<td>1.0021</td>
<td>0.0439</td>
<td>0.078</td>
</tr>
<tr>
<td>0.5</td>
<td>31.586</td>
<td>1.8553</td>
<td>1.0052</td>
<td>0.0219</td>
<td>0.086</td>
</tr>
<tr>
<td>0.2</td>
<td>33.341</td>
<td>2.2921</td>
<td>1.0076</td>
<td>0.0084</td>
<td>0.087</td>
</tr>
<tr>
<td>0.1</td>
<td>34.007</td>
<td>2.6253</td>
<td>1.0083</td>
<td>0.0061</td>
<td>0.084</td>
</tr>
</tbody>
</table>

of cold pressed compacts made from 22 µm copper powder was found to be 10⁴ times higher than compacts made of 3 µm powder. It is coherent since larger particles are more likely to create electric contacts during pressing as reported in [3]. The results for case 2 are presented in Table 2. In both cases an overall tendency is clear that with decreasing particle diameter the value of ε′ increases. In case 1, the value of ε″ is very low and within the error range. Thus, case 1 is not an appropriate model for the early stage of sintering in the electric field only. For case 2 the value of ε″ increases with the decrease in the particle diameter. In both cases the real part of permeability is very close to 1; only for 3 µm particles the value of µ′ indicates that the mixture is diamagnetic. This effect is related to very high density eddy currents which are responsible for the magnetic losses. The value of µ″ follows the same pattern in both cases. The highest value of µ″ is found for 3 µm. The penetration depth of copper powders is in the range of a few centimeters in the case with the oxide layer. It is also significantly larger compared with the penetration depth of the bulk copper δ = 1.34 µm at the frequency of 2.45 GHz. It is noteworthy that even for a very small volume content of oxide layer in case 2, the electric losses are very high. Compared with the value of the imaginary part of permittivity ε″ = 0.9249 for the bulk copper oxide, the electric absorption of the mixture is always higher. One can understand this effect looking at the effective path of the electromagnetic wave in the mixture. Figure 3 shows the cross-sectional plot of power flow for the structure with 2 µm particles. The power flow monitor in CST MWS stores the peak value of the Poynting vector S = E × H. The Poynting vector can be thought of as a representation of the energy flux and indicates the direction of propagation of an electromagnetic wave within the material.

Additionally to the longer effective path of the electromagnetic wave inside the mixture, the electric field which does not penetrate metallic particles is being squeezed between the particles as shown in Figure 4. The effect is known as microfocusing of the electric field, responsible for an enhanced sintering of ceramic materials [19, 20]. The local electric fields are disproportionately intense close to the particle’s surface due to strong focusing. Also, the electric field is much stronger in the inter-particle contact zones and forms spherical neck contacts [21]. Simulations show that the peak value of the microfocused electric field is 10³ times larger than the peak value in free space. High electric field densities in regions where
particles are very close to each other and where the oxide layer is located give rise to relatively high dielectric losses. The high dielectric losses in the contact zones might lead to a quick formation of conductive contacts between the copper particles. This may explain the effect of the initial heating behavior of copper powder compacts heated in the electric field only. The temperature sharply peaks in the initial stage of the heating process and with time the temperature drops and reaches equilibrium. This behavior is observed only during the first heating of the samples [3, 22].

As reported by Ma et al [3], the conductivity of the 3 µm copper powder compact after cold pressing is in the range 0.01–1 S m⁻¹. Using the cavity perturbation methods Ma et al [3] also measured the values of $\varepsilon''$ and $\mu''$ for copper compacts. In the early stage of microwave heating the reported values for $\varepsilon''$ are in the range 0.01–0.1 and values for $\mu''$ in the range 0.001–0.1. The values for $\mu''$ obtained from the simulations are in good agreement with the experimental values. However, the obtained numerical values for $\varepsilon''$ are higher. This may be caused by very high values for $\varepsilon'$ and $\varepsilon''$ used in the simulations for copper oxide, which are close to the value for bulk copper oxide. The thickness of the native oxide layer in copper is in the range of a few nanometers, and the exact dielectric properties are not known.

4. Mechanisms of magnetic losses

Experimentally it has been observed that the heating rate of copper powders, during heating in the magnetic field, resembles the behavior during the heating in the electric field. The temperature sharply peaks in the first few seconds, then drops and reaches a constant value [3, 22]. This behavior was observed only during the initial heating. After cooling and reinserting the sample the sharp peak in the temperature profile does not occur [3].

As mentioned before the magnetic losses occurring in non-magnetic, conductive materials are due to induced eddy currents. Figure 5 shows the cross-sectional plot of current density at the peak value for the structure with 2 µm particles at a frequency of 2.45 GHz. Simulations show that for an alternating magnetic field the eddy current density is the strongest at the surface of the particles. A magnetic field of 200 A m⁻¹ is chosen, which is comparable to the field value in the single mode cavity for an input power of 1800 W. The eddy current density in the region close to the surface of the metallic particle can reach $1.16 \times 10^8$ A m⁻² for a
Figure 4. Cross-sectional plot of the strength through the $z$-planes of the electric field for the structure with $2 \mu m$ particles. The size of the arrows corresponds to the strength of the electric field, with scaling factor of $\log_{100}$ for better representation. The effect of microfocusing is clearly visible, leading to high dielectric losses in the region near to the surface of the particles.

Structure with $2 \mu m$ particles, while the average value of the eddy current density in the near surface region is $7.35 \times 10^7$ A m$^{-2}$. This very high eddy current density leads to considerable power losses at the surface and near the surface region reaching $7.35 \times 10^7$ W m$^{-3}$. Extremely high heating rates explain the quick formation of conductive contacts between particles. The
heating of the copper powder compacts in the magnetic field is characterized by a sharp peak in the temperature at the early stage. This stage corresponds to the surface heating of particles which can be considered as non-interacting absorbers. After the initial high rate of surface heating of the particles the conductive contacts are being formed and the particles cannot be considered as separate absorbers anymore. This leads to a dramatic decrease in the absorption of the microwaves and thus decreased heating rate. After the conductive contacts between particles are formed the heating rate relaxes and reaches a plateau [3, 22].

In order to study the effects of absorption of the magnetic field component, simulations were conducted for spherical particles with diameter 1–10, 15 and 25 μm. The simulations were performed for a wide frequency range from 1 to 20 GHz. The complex values for ε and μ were extracted for a single frequency of 2.45 GHz, and used in later simulations of the microwave heating of the copper powder compacts. Figure 6 shows the real and imaginary parts of the complex magnetic permeability, respectively. One can notice a clearly visible resonance behavior. The resonance frequency depends on the particle size and conductivity of the material. For the sake of simplicity we only present the results for the copper particles. The dependence of the absorption coefficient (μ″) on the particle diameter implies different heating rates of the copper powder compacts composed of particles with different diameters.

5. Simulations of microwave heating in the magnetic field

Microwave heating in the single mode (TE_{102}) cavity was performed using COMSOL multiphysics FEM software. The simulation results are compared with the experimental values of the initial heating rate as presented in [3].

The setup of the model is similar to the one presented in [23]. Figure 7 shows the placement of the copper powder compact sample within the cavity with dimensions of 7.2 cm width, 3.6 cm height and 22.63 cm length. The sample is placed at the maximum of the magnetic energy density close to the cavity wall. The model of the cavity and the sample consists of around 50k mesh elements. The coupled problem is solved in two steps. First, the stationary solution for the electromagnetic waves inside the cavity is obtained. The perfect electric conductor (PEC) boundary condition is set for all walls except the wall where the
microwave port is located as shown in figure 7. The solution is a standing wave with two anti-nodes of the $E$-field component and the $H$-field rotating around the $E$-field anti-nodes. The electromagnetic properties of the sample were taken from the extracted values of $\varepsilon$ and $\mu$ for the copper particles at 2.45 GHz frequency.

The second step is the transient solution of the heating process. The boundary condition for all the cavity walls was set to the constant ambient temperature 300 K. For the surface of the sample the selected boundary condition was a heat sink with radiation type surface to ambient. This type of boundary condition describes the surface radiosity according to the Stefan–Boltzmann law

$$J_0 = \varepsilon_s \sigma (T^4 - T_r^4),$$

(20)

where $J_0$ is the total energy radiated per unit area per unit time, $\varepsilon_s$ the surface emissivity, $T$ the temperature in kelvin, $T_r$ the room temperature and $\sigma$ the Stefan–Boltzmann constant. The value of surface emissivity is the same as for bulk copper, $\varepsilon_s = 0.7$ [23].

The heating time was set to 10 s, then the heating rates were normalized for comparison with the experimental results [24].

![Figure 6. Simulated real (a) and imaginary (b) parts of the magnetic permeability for copper powder compacts for selected particle diameters.](image-url)
Figure 7. The single mode (TE_{102}) cavity as used in simulations of microwave heating in the magnetic field only. The arrows represent the magnetic field. The copper powder compact sample is placed close to the wall, where the magnetic field is the strongest.

Figure 8. Initial heating rate of the copper powder compact. The results of the microwave heating simulations (dotted line with crosses) are compared with the experimental results (dots) taken from [24]. The simulated heating rates are in very good agreement with the experimental data taken from [24] up to the particle diameter of 8 \( \mu \text{m} \) as shown in figure 8. In the further range the simulated heating rates are much bigger than experimentally found. As mentioned in section 3 the bigger particles are more likely to create conductive contacts during the compaction procedure [3]. The simulation does not take this effect into account.

6. Conclusions

A numerical approach for determining effective dielectric and magnetic properties of metallic powders was presented in this work. Similar to the calculation of effective properties of complicated structures of metamaterials the combination of electromagnetic simulations and the extraction of effective properties can be used with success in the field of material science. The simulation results are compared with the experimental data allowing one to study and understand the basic mechanisms of microwave absorption as well as the sintering of metallic
powders. The heating behavior of copper powders in either the electric or the magnetic field is similar; however, it is driven by different mechanisms. During the sintering in the electric field the microfocusing effect plays an important part. The electric field is much stronger in the contact zones between the particles leading to tremendous dielectric losses in the native oxide layer covering the copper particles. During the sintering in the magnetic field the conductive particles are heated by induced eddy currents. The absorption of the magnetic component depends on the conductivity and the particle size. The simulations over a wide range of frequency showed resonant behavior in the effective magnetic permeability of copper powders. While the absorption of the electric component can only be explained qualitatively the absorption of the magnetic component of the microwaves is also quantitatively in good agreement with the experimental results. Further studies must be performed to fully explain and understand the sintering process of the metallic powders. Future simulations should include heating in the microwave field on the microscale as well as the modeling of the neck formation between metallic particles. The evolution in time of the properties of the inter-particle contact zones is essential to make a complete simulation of the sintering process.

Acknowledgments

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