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Measurement of average particle size in metal powders by microwave cavity perturbation in the magnetic field

N. Clark[a,*], N. Jones[b], A. Porch[a]

[a] Centre for High Frequency Engineering, Cardiff University, Cardiff, CF24 3AA, United Kingdom
[b] Renishaw Plc, New Mills, Wotton-under-Edge, Gloucestershire, GL12 8JR, United Kingdom

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ABSTRACT

The magnetic absorption of metallic powders, particularly at microwave frequencies, is of great theoretical and practical interest and has been the subject of previous research examining the dependence of absorption on the ratio of the particle skin depth to radius. Here, the validity of the theoretical approach concerning the peak in the absorption spectrum is verified using a 3D simulation of a hexagonal, close-packed particle matrix. Clear experimental data is given for the real and imaginary parts of the magnetic permeability of metal alloy powders (Ti6Al4V), of varying size, obtained by using the cavity perturbation technique across three separate frequencies in the GHz range. The results are shown to be congruent with existing theory. Further verification of the absorption peak is given by the testing of the powder at lowered conductivity by elevating the temperature. The results demonstrate the applicability of the relatively simple microwave cavity perturbation approach to the determination of the average particle size in a metal powder when compared with other, more complex and time-consuming methods.

1. Introduction

Since the first demonstration of sintering a metal powder body by microwave radiation [1], efforts to understand the energy absorption of conducting metal powders has been ongoing. Whilst the simple need to generate heat has been the primary concern when approaching this subject, there is much value in considering other measurement applications. For instance, from accurate knowledge of the absorption behavior of an unknown metal powder, the approximate mean particle size can be determined, or particle sizes between batches of powder can be compared.

The theoretical basis for electromagnetic absorption in conducting metal powders is well established. A variety of studies have taken a first principles approach to the absorption of an individual particle within both electric and magnetic fields [2–4]. It is accepted that, for any practical size of particles which are considered to be metallic on the basis of a high value of electrical conductivity, magnetic absorption via eddy current loss is much greater than loss in an equivalent electric field and this has been demonstrated experimentally [5,6].

Typically, studies are interested in energy absorption for the application of heating a powder. With this in mind, previous works have been undertaken to experimentally verify the absorption in metallic powders by applying high power microwave radiation and measuring the powder temperature. This has been done relatively successfully by measuring the temperature gradients and maximum temperature achieved [7]. The peak absorption rates, as predicted at specific particle sizes by theory, have also been observed experimentally [8,9]. However, using the temperature as a measure of the absolute absorption has many difficulties. Firstly, the precise power delivered to the sample must be calculated and this may not be trivial due to the complex loading and changing condition of the applicator caused by the heated powder. Also, accurate determination of other powder characteristics will likely be difficult. For instance, the powder thermal conductivity and dissipation rates can be massively affected by the ambient conditions, such as initial temperature and airflow, and other powder characteristics such as packing density, sample size and chemical composition.

Microwave cavity perturbation provides a convenient and potentially easy method of determining the absolute absorption of a powder and, using cavity modes which isolate the electric and magnetic fields at the sample insertion point, magnetic and electric absorption can be measured separately [10–13]. High Q cavities and modern network analyzers ensure that measurement

* Corresponding author.
E-mail address: clarkns@cardiff.ac.uk (N. Clark).

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error is extremely small and modern simulation tools have allowed for accurate correction of errors caused by sample holes and coupling structures [14]. Also, sample preparation is minimal and the technique is flexible to allow measurement at significantly elevated temperature. Another advantage is the use of multiple modes to provide simultaneous measurements at different frequencies [15]. This gives a range of microwave skin depths, which is crucial to determining the mean particle size from magnetic absorption (i.e. due to eddy currents) in metallic particles.

Determination of the particle size distribution for a powdered material is currently a relatively expensive or inconvenient process. In an industrial setting, a survey using scanning electron microscopy (SEM) is usually prohibitively expensive. Laser diffraction is the standard measurement used but this usually requires that the powder is well-dispersed in liquid, a process which in metals is prone to error due to its high density. Furthermore, these systems require significant ongoing maintenance and replenishment of consumables which makes them unattractive for many applications. Conversely, microwave cavity perturbation could provide a convenient method which relies on a simple metal structure for the cavity, and solid-state electronics to interrogate it. Information regarding the average particle size of a metallic powder can then be obtained non-destructively. The information it provides, although not a detailed particle size distribution (PSD), can be useful, for instance, in determining the evolution of powder properties over time.

2. Background

2.1. Theory

The complex magnetic dipole moment for an individual spherical particle is derived as [2]

$$m = 2\pi a^3 H_0 \left(\frac{(\mu+2)\left(1 - ka\cot(ka) - \mu(ka)^2\right)}{(\mu-1)\left(1 - ka\cot(ka) - \mu(ka)^2\right)} \right) \quad (1)$$

where a is the particle radius, H_0 is the applied magnetic field strength, ε is the internal sphere permittivity, μ is the internal sphere permeability and the wavenumber $k = \frac{\omega\sqrt{\varepsilon\mu}}{c}$. Electrical conductivity σ is introduced by making ε complex, i.e. by considering $\varepsilon = \varepsilon_1 - j\sigma/\omega\varepsilon_0$, where the imaginary part dominates the real part ε_1 for materials considered to be weakly conducting, and certainly so for materials considered to be metallic.

This leads to simplified expressions for the power absorbed per unit volume and the real part of the relative permeability for a non-magnetic conducting powder as

$$\langle P_M \rangle = \frac{3}{4}\omega\mu_0 H_0^2 \mathrm{Im}\left(1 + \frac{3\cot(ka)}{ka} - \frac{3}{(ka)^2}\right) \quad (2)$$

$$\mu_1 = 1 - \frac{1}{2}\mathrm{Re}\left(1 + \frac{3\cot(ka)}{ka} - \frac{3}{(ka)^2}\right) \quad (3)$$

Considering the large skin depth limit, $a/\delta \ll 1$ we see that for small particles

$$\lim_{\frac{a}{\delta}\to 0} \langle P_M \rangle = \frac{1}{20}\omega^2\mu_0^2 a^2 \sigma H_0^2 \propto \omega^2 a^2 \sigma \quad (4)$$

Conversely, in the small skin depth limit, we see that for large particles

$$\lim_{\frac{a}{\delta}\to \infty} \langle P_M \rangle = \frac{9}{4a}\sqrt{\frac{\omega\mu_0^2}{2\sigma}} H_0^2 \propto \frac{1}{a}\sqrt{\frac{\omega}{\sigma}} \quad (5)$$

As can be seen, for fixed value of frequency, the absorption in large particles is inversely proportional to the particle radius and

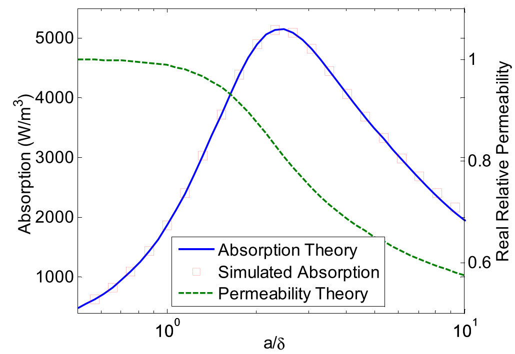

Fig. 1. Comparison of simulation and theory of the absorption and permeability of a single spherical particle. Theory curves are given by Eqs. (2) and (3).

proportional to the square of the radius for small particles. This reveals an absorption peak which is found when the radius is 2.4 times as large as the skin depth.

The above results are for isolated spheres, so a key assumption is that the particles are arranged suitably sparsely such that local magnetic field corrections caused by particle-particle interactions can be ignored. However, meeting this criterion in reality is difficult due to the nature of the powder itself. Attempts to suspend the powder in some type of setting liquid are unlikely to be successful due to the high density of the individual metal particles, thus leading to particle settling. The relevance of the equations is therefore in question but this can be answered, to some extent, with simplified simulations.

2.2. Simulation

Simulations were undertaken using COMSOL Multiphysics at 2.45 GHz using a quasi-static approximation. A uniform magnetic field of amplitude 1 A/m was applied in a region of space with boundaries kept suitably far away from the particles such that the magnetic field at the boundary is unaffected by local field corrections owing to the particles. This condition mirrors the assumption made when applying first order perturbation theory. The simulations are not intended to completely characterize the absorption for multi-particle systems but simply give an indication of their behavior. In order to characterize the absorption empirically, many different packing schemes would need to be considered as well as various field orientations. In the first instance and for this case, a simple hexagonal close packed (HCP) system was used as it gives a relative packing density close to what was measured during experimentation (~0.6). The particle radius is fixed at 20 μm and the electrical size is changed by adjusting its conductivity – thus changing the ratio of skin depth to particle radius. Initially a single particle was simulated to verify future simulations, as shown in Fig. 1. The simulation shows very good agreement with the analytical theory, demonstrating the characteristic absorption peak when a = 2.4δ. For completeness, the theoretical real permeability is shown, even though this was not calculated by simulation.

The next 3D simulation considers a hexagonally close packed matrix of 216 particles (i.e. a 6 × 6 × 6 array). The generated particle agglomeration can be seen in Fig. 2. Note the axis directions, which are referred to later. The HCP planes are stacked in the z direction.

Four distinct conditions are considered: the field applied in either x or z direction and with the particles either touching or with a 0.1 μm gap separating all particles. The z direction indicates field application perpendicular to the 6 HCP planes. Fig. 3 shows the result of these simulations with the touching a separated cases showing distinct results. With small inter-particle separation, the measured absorption closely resembles the profile predicted by the