ELECTROSTATIC DETECTION OF DENSITY VARIATIONS IN GREEN-STATE POWDER METALLURGY COMPACTS

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ABSTRACT. Producing P/M compacts is generally a low-cost, high-volume manufacturing effort with very special quality assurance requirements. When considering the three basic P/M steps of mixing, compacting, and sintering, it is the compaction process producing the green-state parts that offer the highest pay-off for quality control through nondestructive evaluation (NDE) techniques. A detection of compacting-related problems in the green-state samples permits early process intervention, and thus prevents the creation of potentially significant numbers of faulty parts. Work at WPI currently has concentrated on extending the previously developed method for crack detections to measure density variations within the parts. In this paper a physical model and a mathematical formulation are reported that are capable of relating green-state density to electric conductivity for various lubricant concentrations. Electrostatic measurements of cylindrical compacts have so far confirmed the theoretical model assumptions. Specifically, the green-state conductivity increases as the sample density increases up to approximately 6.9 - 7.0 g/ccm. Any further density increase results in a decrease in conductivity. Preliminary measurements with a range of cylindrical samples support the theoretical model.

INTRODUCTION

Producing P/M compacts is generally a low-cost, high-volume manufacturing method with very special quality assurance requirements. When considering the three basic P/M steps of mixing, compacting, and sintering, it is the compaction process producing the green-state parts that offer the highest pay-off for quality control through nondestructive evaluation (NDE) techniques. Detection of compacting-related problems in the green-state would permit early process intervention, and thus prevent the creation of potentially significant numbers of faulty parts prior to sintering.

Initial research targeting resistivity measurements of pre-sintered, green-state powder metal (P/M) compacts resulted in a surface-breaking and subsurface crack detection instrument [1-3]. Our current work extends the previously developed approach of evaluating large variations in conductivity over microscopic distances by evaluating small changes of conductivity over macroscopic distances. It is reasoned that the same electrostatic approach should be applicable for the detection of density gradients in green-state compacts. During the course of this density measurement program for green state PM parts, an initial effort was made to link the density of the part to a specific conductivity. This conductivity - density relationship can subsequently be used in the reconstruction of a density map of the part. Interestingly, the outcome of these measurements differed significantly from the expected...
results. For non-lubricated parts, a linear relationship between the nominal density and the conductivity could be established. For lubricated parts, however, not only was the relationship nonlinear, but it also exhibited saturation and even an inversion at high densities [4,5]. Figure 1 demonstrates the inversion of the relationship at high densities for lubricated P/M samples.

Initial attempts to explain the inversion focused on process effects. Specifically, lamination, lubricant migration, and increased oxidation through higher temperatures were thought to be responsible for this response. However, no clear experimental evidence could be developed to support any of these assumptions. As a consequence, further considerations were given to the theory of conductivity of mixtures and answers were found that seemed to explain the initially puzzling effects through physical laws. The following paragraphs attempt to explain the inversion as a physical phenomenon involving induced charges and polarization effects.

NON-CONDUCTING PARTICLES IN CONDUCTING MEDIUM

The problem of determining the electrical conductivity and the dielectric constants are closely related. Both cases result in almost identical Laplace-type equations, where an electric potential must be continuous across the interface of adjacent regions with different material parameters. The required boundary condition arises from the continuity of the electric current density $J = \sigma E$ in the conductive case and the displacement $D = \varepsilon E$ in the dielectric case. Since the field $E$ plays the same role in both cases, the governing equations for the conductivity $\sigma$ and the dielectric constant $\varepsilon$ become identical.

If the non-conducting particles in a conducting medium are treated like molecules in a solid or liquid dielectric spaced in such a way that we can assume that the effect of their presence does not considerably alter the electric field acting on the other particles, then the Clausius-Mossotti equation applies:

$$\varepsilon E_{\text{eff}} - \varepsilon_0 E_0 = \frac{4\pi}{3} \varepsilon \rho = \frac{4\pi}{3} n LE_{\text{eff}}$$ (1)
This equation calculates the summed effect of all particles within the volume on the external electric field \( E_0 \), resulting in an effective field \( E_{\text{eff}} \). Here \( P \) is the macroscopic polarization vector, \( n \) is the particle concentration, and \( L \) represents the depolarization factor. Such a model appears to be a plausible explanation, since the lubricant can be considered as individual particles embedded in the green-state PM base material. Because the lubricant concentration is generally low, on the order of 5% or less, the mutual interaction between the polarizable particles can be neglected. The sum of their effects, however, still leads to an effective field that differs from the applied field.

The idea of having two separate material constituents with different electric properties can also be treated from a purely material’s point of view. Considering a medium consisting of two (or more) constituents with conductivities \( \sigma_1 \) and \( \sigma_2 \) and volume fractions \( f_1 \) and \( f_2 \), on a completely symmetrical basis, leads to Bruggeman’s symmetric theory [7,8]. This theory allows us to calculate the conductivity of a random mixture of spherical particles of two constituents, both of which completely fill the medium. Generalizing the equation to three dimensions \( d=1,2,3 \), the conductivity of medium \( \sigma_m \) is thus given by the equation

\[
\frac{f_1}{\sigma_1+(d-1)\sigma_m} + \frac{f_2}{\sigma_2+(d-1)\sigma_m} = 0.
\]

Using the case of a non-conducting dispersion material ( \( \sigma_1 = 0 \) ) in a highly conductive host medium with \( \sigma_2 = \sigma_h \), Eq.(2) is solved for the conductivity of the resulting mixture as

\[
\sigma_m = \sigma_h \left( 1 - \frac{f_d}{d-1} \right) = \sigma_h \left( 1 - \frac{f_c}{f_c} \right).
\]

Here \( f_c \) denotes the critical insulator volume fraction at which the conductor - insulator transition occurs. In three dimensions \( f_c \) becomes 2/3.

The volume fraction of each constituent can easily be calculated from the known densities of the two constituents and the mixture. Using

\[
g = V \rho,
\]

with \( V \) being the volume and \( \rho \) the specific weight, we can calculate the contribution of each part to the weight of the mixture. Setting these weights into relation with each other, the volume fractions of either constituent can be determined as

\[
f_1 = \frac{V_1}{V_{\text{mix}}} = \frac{\rho_2 - \rho_{\text{mix}}}{\rho_2 - \rho_1}.
\]

The conductivity of a medium, where the dispersion with conductivity \( \sigma_1 \) consists of an effectively infinite size range of spheres, each of which remains coated at all volume fractions with the host medium of conductivity \( \sigma_h \), can be calculated using Bruggeman’s asymmetric theory [8,10]. Using the more general approach of oriented ellipsoids instead of spheres, the equation can be written as [13]

\[
\frac{(\sigma_m - \sigma_d)^{1/L}}{\sigma_m} = (1-f)^{1/L} \left( \frac{\sigma_m - \sigma_h}{\sigma_h} \right)^{1/L},
\]

with \( L \) denoting the depolarization factor of the ellipsoids with the short axis oriented in the direction of the current flow. When the dispersion can be regarded as an insulator compared to the conductivity of the host medium ( \( \sigma_1 = 0 \) ), equation (6) becomes

\[1317\]
Modeling the conductivity of a green state P/M compact was accomplished by calculating the volume fraction of air and lubricant at each density. Using Eq. (7), the conductivity for non-lubricated parts was calculated with the conductivity iron as the base material. In a next step, the resulting conductivity was used as the background conductivity in the calculation of the lubricated parts, resulting in an overall equation of

$$\sigma_{PM} = \sigma_{Fe} (1 - f_{air})^{1/\lambda}$$

Using these parameters to simulate the conductivity – density relationship over a wide density range does not show the results we obtained during the experiments. Although the volume fractions for the air and the lubricant account for a reduction in the overall conductivity, the relationship is still linear. This is explained by the fact that the volume fraction of the lubricant stays constant through the compaction process. The increased amounts of non-conducting lubricant particles per volume at high densities only results in a lower slope in the still linear relationship and cannot explain the inversion behavior.

**DEPOLARIZATION EFFECT**

Up to this point the lubricant particles and the air inclusions within the pressed part were regarded as perfect spheres. As seen above, the linear increase of low-conducting particles per volume with a linear increase in density leads to a linearly increasing conductivity, with the amount of lubricant in the mixture determining the slope of the relationship. Instead of regarding the depolarizing particles as constant spheres, we now change this viewpoint and take the geometrical deformation of the lubricant into account.

The depolarization factor, which enters equation (7), depends on the geometry of the embedded particles. Let us consider an ellipsoidal particle of uniform dielectric properties, which is bounded by a surface defined by

$$f(x, y, z) = \left(\frac{x}{a}\right)^2 + \left(\frac{y}{b}\right)^2 + \left(\frac{z}{c}\right)^2.$$  

In the presence of a uniform external field $E_0$, the interior field is given by the superposition of the external field and the depolarization field $E_1$, which is produced by a surface charge. The depolarization potential at any point within the ellipsoid is given by the surface integral

$$\Phi(r') = \frac{1}{4\pi \varepsilon_0} \oint_{S} \frac{P \cdot n}{r} dS,$$  

where $P = \varepsilon E$ is the polarization of the dielectric. Since the depolarization field inside the ellipsoid is uniform [12], we can determine it at any point. The center of the ellipsoid is the obvious point to choose. Here the exterior electric field is given by

$$E_i(r') = E_i(0) = -\frac{1}{4\pi \varepsilon_0} \int \frac{P \cdot n}{r} r dS = -\left(\frac{P_x x}{a^2} + \frac{P_y y}{b^2} + \frac{P_z z}{c^2}\right)d\Omega.$$  

The depolarization factors, which are usually defined by
can now be calculated by rewriting (11) in polar coordinates \[6\] in the form

\[
L = \frac{1}{e^2} \int_0^{2\pi} \int_0^{\pi/2} \sin \vartheta \cos^2 \vartheta \left( \frac{\sin^2 \vartheta \cos \varphi}{a^2} - \frac{\sin^2 \vartheta \sin \varphi}{b^2} + \frac{\cos^2 \vartheta \cos \varphi}{c^2} \right) d\vartheta d\varphi.
\]

(13)

For a spheroidal geometry \((a = b \neq c)\) this integral can easily be evaluated. If the external field is applied along the z-axis the depolarization factor for the oblate spheroid of results in

\[
L_z = \frac{4\pi}{e^2} \left( 1 - \frac{\sqrt{1-e^2}}{e} \right).
\]

(14)

where \(e = \sqrt{1-(c^2/a^2)}\) refers to the ellipticity of the rotated ellipse with \(c\) denoting the short half-axis, and \(a\) the long half-axis of the oblate spheroid.

The depolarization factor clearly changes in non-linear fashion. Starting from a perfect sphere with \(L_z = 1/3\), a linear change of the dimension in z direction results in a non-linear change of the depolarization factor and approaches the numerical value of 1 when the extension in z-direction approaches zero.

If we again use (7) as the basis for calculating the conductivity of the PM samples, the depolarization factor remains no longer constant. Increasing the density of the parts will result in a deformation of the lubricant particles. Since the lubricant itself is incompressible and can only change the shape, the volume of each lubricant particle is assumed constant. Furthermore, assuming that the spheroidal properties of the particle are conserved, any compression in z-direction results in a reduction of the short half axis \(c\) and a corresponding increase of the longer half axis \(a\). This enlargement of the cross-section perpendicular to the current flow enhances the non-linear effect of the depolarization factor with increasing density. The sharp increase of this depolarization factor ultimately leads to a highly increased depolarization effect at high densities. Therefore the voltages recorded over a given length, while injecting a constant DC current, begin to increase instead of decrease. This, in turn, leads to the experimentally observed decrease in conductivity at high densities. Figure 2

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**FIGURE 2.** Simulation of conductivity vs. density behavior for green state PM parts with different amount of lubricants.
shows the result of the simulations, where the conductivity was calculated for non-lubricated parts and for three different lubricant mixtures. The inversion can clearly be observed.

INITIAL DENSITY RECONSTRUCTION MEASUREMENTS

Initial experiments were made to reconstruct density gradients from cylindrical samples. The green parts used in the measurement were 2.75" to 3.25" inches long and all had a radius of 0.75", which corresponds to an aspect ratio that promotes large density gradients throughout the part. Using the same powder mixtures that were used to press the flat disks to measure the density-conductivity relationship, the green samples had the following properties:

- iron 1000B as base material,
- four different sets of parts with no lubricant (die wall lubricated), 0.3% AWX, 0.5% AWX and 0.75% AWX respectively,
- each set with 7 to 8 parts of different pressures, ranging from 25 tsi to 55 tsi
- single punch pressing (to increase density gradient)

The results of the voltage measurement over the surface of the long, thin green state P/M rods show a distinct change in conductivity over the length of the part. Even with the limited resolution of 0.25" for our first measurements, a conductivity, and hence a density distribution, is apparent.

Together with the previously recorded relationship between the conductivity of pressed powder and its density, the density distribution over the length of the parts can be reconstructed. This reconstruction is straightforward in the case of non-lubricated parts, where the relationship is linear and each conductivity can be directly mapped into a corresponding density over the entire range of measurements. The densities for the lubricated parts are reconstructed in much the same way, with some intelligence added to the algorithm to discriminate between densities above and below the inversion point. Such an algorithm not only takes into account the voltage reading for the inspected segment, but also the readings from the neighboring segments in order to determine the general direction of the density gradient in the vicinity.

In order to verify the validity of the density distribution obtained from the conductivity measurements, destructive density measurements were performed on the previously analyzed samples. The samples were cut into 0.25" thick slices, exactly the same spacing used for the non-destructive density reconstruction. These slices were then weighed on a precision balance and average density for each slice was calculated.

Figure 3 compares the density values that were reconstructed from our conductivity measurements to the actual density values obtained from the sliced samples. Not surprisingly, the numbers do not match exactly, but the correlation between the data sets recorded with the two different methods is striking. Several interesting observations can be made explaining the presented data:

- It is obvious that whenever the real density (as measured on the sliced samples) falls below 6.0 g/cm³, our reconstruction begins to fail. This is true for both lubricated and non-lubricated parts. This is attributed to the fact that our conductivity-density baseline does not contain data below this density point. Apparently the extrapolation beyond this point as assumed for the reconstruction at hand is not valid. Establishing an extended baseline would solve this problem.
For densities above 6.0 g/cm³ the reconstructed density is close to the actual values found for the sliced samples. Even more striking is the similarity of the density changes over the length of the parts. This observation stresses once more the fact that our method delivers reasonable absolute numbers. The real value of the method, however, lies in its comparative measurements against an established baseline. It is not the goal of this method to accurately measure absolute density values, but to detect variations of density within parts and from one part to the next.

**FIGURE 3.** Comparison of density values obtained from predictive and destructive testing respectively. Results shown are for green state PM parts made from 1000B powder with 0.75% AWX lubricant at three different nominal pressures: a) 25 tsi, b) 40.8 tsi, c) 55 tsi.
CONCLUSION

A new electrostatic model is presented that is capable of predicting the conductivity in green state compacts. Preliminary simulations show very good agreement between this model and actual measurements. Several different model parameters are still available to adjust the simulation results including the degree of deformation of the lubricant particles, the density at which lubricant deformation begins to contribute, and the rate of deformation with increasing density. In a similar sense, one could also argue that the air bubbles within the part will be deformed to ellipsoidal shape prior to being forced out of the compressed powder. Such a deformation would increase the depolarizing effect of these air bubbles much in the same way as the deformation of the lubricant does. However, because of the good agreement with measurements, no such modifications of the present model are needed.

The aforementioned parameters are likely to change with each base material, lubricant, and even lubricant amount. The exact relationship is therefore difficult, if not impossible, to predict a priori without certain baseline measurements. Nonetheless, the presented model sufficiently explains the reasons for the inversion of the conductivity – density relationship at high densities for lubricated PM parts, a key feature arising in the electrostatic green state material characterization.

The reconstruction of density variation in cylindrical parts using the established conductivity-density relationship was successful. Destructive control measurements validate the results found by the new method. Additional work, currently under investigation at WPI, is required to extend the method’s capabilities to complex sample shapes.

REFERENCES

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