Simulations for Powder Materials and Production Process Design

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Introduction

Owing to advances in powder simulation, exponential progress is being made in powder processing operations and in the design level of powder processes, but as process design becomes more sophisticated, it is necessary to set forth the design conditions (objectives) of a process more quantitatively and definitely than before, which is creating new challenges. Let us explain this problem with the example of designing a manufacturing process for functional ceramic materials.

Functional materials are also known as energy conversion materials because when mechanical, electromagnetic, thermal, optical, or chemical energy is applied to them, they convert it into a different form of energy. One well-known type of functional ceramic material is barium titanate ceramics, which convert applied mechanical energy to electrical energy because they are piezoelectric.

Barium titanate ceramics' permittivity and piezoelectric characteristics basically arise from spontaneous polarization that occurs owing to the perovskite crystal structure of BайTі0₃. But because that ceramic material is a kind of composite material comprising the three phases of crystal grains, grain boundaries, and pores, the material texture makes for great changes in the characteristics of the ceramic material. Hence the BайTі0₃ ceramics manufacturing process creates a constitutive texture (microstructure) that is optimum for providing the ceramics with the desired permittivity and piezoelectric properties through the three processes of powder raw material processing, forming, and sintering.

Accordingly, it is necessary in designing ceramic manufacturing processes to fully understand the relationship of ceramic materials' permittivity and piezoelectric properties to the materials' microstructure, and to have a clearly defined microstructure for the ceramics that are to be made in that manufacturing process. Even now, however, there is hardly an instance in which the relationship between ceramic material characteristics and microstructure is quantitatively known, and it is impossible to set forth a quantitative and definite ultimate objective for the design of processes whose main purpose is optimum microstructure formation.

Powder engineering is an academic discipline concerned with the creation of materials in which powders are involved, and the design of processes to bring that about. This discipline must for the next century hurry to establish an engineering system that covers powder materials from design to production. In doing so, powder simulation will play a very great role, and simulation will certainly serve as a tool in achieving these aims.

This paper shall use BайTі0₃ ceramic materials as a specific example in giving an overview of the use of powder simulation in the microstructure design and process design of powder materials.

1. Powder Simulation

For some time people have insisted on the need to organically use the three methods of experimentation, theory, and simulation (Fig. 1) in order to understand natural phenomena. This is especially impor-

![Fig. 1 Understanding of Powder Phenomena (the role of simulations)](image-url)
tant for the understanding and engineering analysis of powder phenomena, which appear complex because many parameters are intricately involved. But as powders are aggregates of many solid particles, their behavior exhibits a strong tendency for dispersing. This makes it difficult to apply simulation methods based on continuum dynamics, which has seriously delayed the application of simulations to powders. But the amazing advances in computers over the last 10-odd years now allow individuals to easily use workstations with powerful calculating capacities, and that makes it possible to perform not only continuum dynamics simulations, but also discrete element simulations such as those based on molecular dynamics. Inspired by this, discrete element simulations have been developed for the behavior of powders, which have a strong dispersive nature, which has catalyzed vigorous efforts to simulate powder phenomena and realized considerable achievements.

Currently researchers use a variety of powder simulation methods including molecular simulations, continuum dynamics simulations, and discrete element simulations, as well as hybrid simulations that combine these, for the purposes of designing the desired powder particles and materials, and analyzing powder behavior.

2. Microstructure Design of Ceramic Materials [1]

In the design of ceramic manufacturing processes, it is necessary to design microstructures according to the law of mixtures, which indicates the relationship between the characteristics and microstructures of ceramic materials, as noted above, and to design unit operations and processes. Thus as an example of simulation for microstructure design, we shall use a simulation to elucidate the relationship between permittivity and microstructure in barium titanate (BaTiO₃), and show that the microstructure design of dielectric ceramics is possible.

2.1 Permittivity Estimation Model

The microstructure of sintered barium titanate varies considerably depending on powder material characteristics, whether or not binders are used, and forming or sintering conditions, with the resulting microstructure greatly affecting permittivity. It being difficult to experimentally determine the relationship between permittivity and the divers changes in microstructure, a prediction method based on an appropriate estimation model is necessary. The equivalent circuit model has long been used to represent the electrical characteristics of ceramic materials [2, 3]. We therefore propose a permittivity estimation method that applies this equivalent circuit model and gives permittivity, sizes of crystal grains, pores, and grain boundaries in the sintered structure, and how their distributions are related.

2.1.1 Unit Cell: A Constituent Element of Microstructure

As shown in Fig. 2, the sintered structure consists of grains, grain boundaries, and pores. We shall consider as the constituent elements of this structure the unit cell, which as shown in Fig. 3(a) comprises grains that are the ferroelectric phase, grain boundaries that are the paraelectric phase, and pores that are the air phase. As shown in Fig. 3(b), this unit cell is stacked three dimensionally, and then the sizes of the grains and pores and the grain boundary thickness in the unit cells are juxtaposed against the sizes of the grains and pores and the grain boundary thickness, as well as their distributions, in an actual sintered product, thereby representing the sintered product's microstructure. Because the unit cells are stacked on one another, the grain boundaries that surround the cells are placed along three surfaces of each cell as shown in Fig. 4(a), whereby the grain boundary effects in the cells are concentrated on these three faces. These unit cells are an improvement over the

![SEM photograph of microstructure in BaTiO₃ ceramics](image1)

(a) SEM photograph of microstructure in BaTiO₃ ceramics

![Rough sketch of microstructure](image2)

(b) Rough sketch of microstructure

Fig. 2 Microstructure of Ceramic Materials
so-called cubes model [4, 5], and their equivalent circuit appears in Fig. 4(b). In this circuit there are three circuits connected in parallel: (1) the grain boundary alone, (2) the grain and boundary connected in series, and (3) the pore, grain, and boundary connected in series. If the resistance $R$ of each part is assumed to be infinite [3], the permittivity of a unit cell can be derived as follows.

Generally when there is a substance in which $k$ phases, from phase 1 to phase $k$, whose volume ratios and permittivities are, respectively, $v_1, v_2, ..., v_k$ and $\varepsilon_1, \varepsilon_2, ..., \varepsilon_k$, are connected in series or parallel, the entire substance's permittivity $\varepsilon$ is indicated by these equations.

**a) Series**

\[
\varepsilon_s = \frac{1}{\sum_{i=1}^{k} \varepsilon_i \cdot v_i}
\]

**b) Parallel**

\[
\varepsilon_p = \sum_{i=1}^{k} \varepsilon_i \cdot v_i
\]

Then if grain size is $d$, pore size is $a$, grain boundary thickness is $t$, and their permittivities are $\varepsilon_g$, $\varepsilon_p$, and $\varepsilon_a$, respectively, the permittivity $\varepsilon_{pg}$ of the portion comprising grain and pore combined in Fig. 3(a) is given by the following equation.

\[
\varepsilon_{pg} = \varepsilon_g \left( \frac{a^2}{d} \frac{\varepsilon_p}{d-a} \varepsilon_s + \frac{a}{d} \right) + \left( 1 - \frac{a}{d} \right)^2
\]

The grain and pore portion is connected to the grain boundary in series on its bottom and in parallel on its side. In consideration of this, the following equation gives the permittivity of the unit cell as a whole.

\[
\varepsilon = \left[ 1 + \frac{d}{t} \frac{\varepsilon_{pg} \varepsilon_s (d+t)}{\varepsilon_d t + t \varepsilon_{pg} (2d+t)} \right]^{1/2}
\]

### 2.1.2 Sintered Product Permittivity

In a unit cell stack as shown in Fig. 3(a) the sizes of grains and pores and the thickness of boundaries differ from one cell to the next. Thus the geometric size of each unit cell would ordinarily be different, but in order to facilitate cell stacking this model assumes that individual cells each having their own unique characteristics are incorporated into a cell whose geometric size is uniform, and that each individual cell is interlinked with its surrounding cells. If a large enough number of cells is used to estimate permittiv-
ity, the error due to this stacking method is thought to be small.

If we create a cube in which the numbers of cells \( l \), \( m \), and \( n \) are arranged along axes \( x \), \( y \), and \( z \) as in Fig. 3(b), the permittivity against an electric field in the \( z \) direction of the entire cubical stack is given by Eq. (6). In other words, in the \( z \) direction \( n \) unit cells are connected in series, and \( 1 \times m \) cell rows connected in series are connected with one another in parallel. Here the permittivity \( \varepsilon_{ij} \) of the cell row connected in series in the \( z \) direction \((i, j)\) is given by this equation.

\[
\varepsilon_{ij} = \prod_{k=1}^{n} \varepsilon_{ijk} = \frac{1}{\prod_{k=1}^{n} \varepsilon_{ij(k)} + \sum_{k=1}^{n} \frac{\varepsilon_{ij(k)} - \varepsilon_{ij(k)}}{\sum_{k=1}^{n} \varepsilon_{ij(k)}}}
\]  (5)

Accordingly, the permittivity of the entire cell stack \( \varepsilon_T \) connected in parallel with cell row \((i, j)\) is given by this equation.

\[
\varepsilon_T = \sum_{i,j} \varepsilon_{ij} \cdot \varepsilon_{ij}
\]  (6)

### 2.1.3 Estimating Permittivity

As crystal particle distributions obtained through experimentation generally have a normal logarithmic distribution, we decided that the particle size of each unit cell would assume a normal logarithmic distribution using random numbers and based on the average, minimum, and maximum particle sizes \( d_{av} \), \( d_{min} \), and \( d_{max} \), and on the standard geometric deviation \( \sigma_g \). Similarly for pore size as well, we randomly placed pores of varying sizes within each unit cell based on pore size distribution and relative density.

Particle permittivity, meanwhile, depends on particle size. We therefore established the permittivity corresponding to each particle diameter based on the relationship between particle size and permittivity reported by Arlt et al. [6]. Additionally, as Takeuchi et al. [3] report that sintered BaTiO\(_3\) has a boundary thickness of 4 nm and permittivity of 100, we used those values in the calculations below.

### 2.2 Permittivity According to the Estimation Model

Fig. 5 shows the experimental results for the relative density and permittivity of BaTiO\(_3\) ceramics. In this experiment a sintered product whose relative density was under 93% had an average grain size of about 1 \( \mu \)m, standard geometric deviation \( \sigma_g \) of 1.4, and average pore size of 0.012 \( \mu \)m. The solid curve in the graph represents the relationship between permittivity and relative density, which was calculated by varying the pore ratio on the basis of the values given above. Similarly, Figs. 6 and 7 show the relationship of average grain size and its distribution to permittivity. These three examples of calculated values all conform closely to calculated results, which indicates that the estimation model proposed above can estimate...
the permittivity of sintered BaTiO$_3$.

As indicated by Fig. 5, if one performs fine-textured sintering without inducing an increase in grain size, it is possible for the sintered product to achieve a permittivity of about 5800, and to gain detailed knowledge about the effects of boundary and pore characteristics, as shown in Fig. 8. While this is but one example, this permittivity simulation provides detailed information about the relationship between the microstructure and permittivity of ceramics, and makes it possible to quantitatively design a microstructure in order to obtain the desired permittivity.

3. Sintering Behavior Simulation

Once one determines the microstructure of ceramics with the desired characteristics, it is necessary to design a process that will unfailingly deliver that microstructure. For that reason we shall next perform a simulation of the sintering phenomenon, which has a major influence on the formation of ceramic microstructures, and elucidate the relationship between sintering behavior and operating conditions.

3.1 The Sintering Phenomenon [7]

When placing two solid particles in a temperature field below the melting point of the solid, a neck forms between the particles as shown in Fig. 9, and the particles' surface areas are reduced. This phenomenon is called sintering, and it is a basic and important phenomenon in making ceramic materials.

Much research has been performed on this sintering mechanism for many years. A neck is formed by the dispersive movement mechanism of atoms as illustrated in Fig. 10. Many sintering speed equations

![Fig. 8 Effects of Grain Boundary Thickness and Pore Size on Permittivity](image)

![Fig. 9 Initial Sintering Behavior of Cu Particles](image)

Table 3.1

| Material movement mechanism | Route                      |
|----------------------------|----------------------------|
| 1. Evaporation-condensation| Surface $\rightarrow$ neck  |
| 2. Dissolution-precipitation| Surface $\rightarrow$ neck  |
| 3. Surface diffusion       | Surface $\rightarrow$ neck  |
| 4. Volume diffusion        | Surface $\rightarrow$ neck  |
| 5. Boundary diffusion      | Grain boundary $\rightarrow$ neck |
| 6. Volume diffusion        | Grain boundary $\rightarrow$ neck |
| 7. Volume diffusion        | Transposition $\rightarrow$ neck |
| 8. Flowing                 | Bulk $\rightarrow$ neck     |

![Fig. 10 Atomic Movement Mechanism During Initial Sintering Phase](image)
have been proposed in correspondence to the dispersion paths of atoms and molecules, but sintering never proceeds under a single mechanism, instead always requiring a complex of two or more. It is therefore difficult to obtain guidelines for actual sintering operations from the sintering speed equations now proposed.

For this reason we carried out a computer simulation of the composite sintering behavior of particles. Material movement occurring in the initial stage of the sintering process is conceived as two separate processes, agglomeration and densification. The agglomeration process is a material movement mechanism involving no change in the distance between the centers of two particles (evaporation-condensation, surface dispersion, surface volume dispersion), and densification is the process by which atoms move according to a mechanism that reduces the distance between the centers of two particles (grain boundary dispersion, grain boundary volume dispersion). If one assumes that the volume of atoms that move in the condensation and densification processes is preserved in the neck, and that sintering time is quantized by microtime \( \Delta t \), and then repeatedly calculates the amount of atomic movement in the condensation and densification processes, it is possible to simulate a composite sintering process.

**Fig. 11** compares experimental and simulation results for the relationship between neck radius and sintering time. Nearly all the experimental values fall within the range predicted by calculations, showing that simulation results were excellent. **Fig. 12** shows the quantities of atoms that moved in accordance with the various movement mechanisms. This simulation provided detailed information on sintering conditions and atomic movement mechanisms.

### 3.2 Microstructure Formation [8]

Large numbers of powder particles are involved in the formation of ceramic microstructures, and many parameters such as sintering temperature, temperature increase rate, and cooling rate also contribute to the outcome. We therefore used the Monte Carlo method in conducting a simulation of microstructure formation behavior, and examined the relationship of microstructure formation to the powder characteristics and sintering conditions of ceramic materials. The simulation principle is as follows.

Triangular lattices are placed on a two-dimensional coordinate system, and the lattice aggregation used to represent grains. We provided interface energy corresponding to boundary-boundary, boundary-grain interior, and interior-interior interfaces, and also took into account anisotropy relating to grain growth. To begin with, the simulation randomly selected and moved one lattice in the sintered product, and used the following Monte Carlo algorithm on the pre-/post-movement energy difference.

When the system's potential energy \( U_i \) becomes \( U_j \) due to movement, state \( j \) is adopted if (i) \( U_j < U_i \). (ii) If \( U_j > U_i \), a uniform random number \( \eta \) between 0 and 1 is generated; if (a), state \( j \) is adopted, but if (b), movement is not recognized. Here \( k_B \) is the Boltzmann constant and \( T \) is absolute zero.

**Fig. 13** shows the change in microstructure when a Ni plate is heated. By contrast, Monte Carlo simulation results appear in **Fig. 14**. Both show that grains grow as sintering time lengthens (Monte Carlo step). Although the Monte Carlo simulation does not include the concept of time, a little innovation makes it possible to consider the correspondence with sintering time. **Fig. 15** graphs the relationship between average grain size \( D_{50} \) and sintering time, and **Fig. 16**...
show the standard geometric deviation \( \sigma_g \) of grain size distribution. As experimental and simulation results closely coincide, it is evident that a Monte Carlo simulation can examine the relationship between microstructure formation and sintering conditions.

4. Designing Powder Operations

The ceramic manufacturing process includes powder operations such as the powder material processing and formation before sintering operations, thereby making powder flow simulation necessary. Powder simulation methods are the (1) finite difference method (FDM), (2) finite element method (FEM), and particle element method (PEM), also known as the distinct element method (DEM). Especially representative are FEM and PEM.

4.1 PEM Simulation of Powder Compression Formation Behavior [9]

When small-particle powders are packed very densely and particles move little, as in powder compression, it is possible to use FEM, a continuum dynamics simulation. Using FEM necessitates three basic equations: (1) a stress equilibrium equation, (2) a conformity conditions equation, and (3) a composition equation. Generally (1) and (2) are easily obtained, but the difficulty of finding the composition equation, which shows the relationship between a powder's state of existence and its dynamic nature, calls for resourcefulness.

For powder compression formation, the example given here, we obtain the composition equation by assuming that the powder bed is an elastoplastic continuum. Additionally, the dynamic characteristics of the powder bed interior, where compression increases, change from moment to moment. Then we indicate the powder's interior friction coefficient by the bulk density function, and Young's modulus and strain...
hardening rate by the minimum principal stress function, thereby making it possible to consider the moment-by-moment state change that happens as powder compression proceeds.

Fig. 17 is the stress-distortion curve in the powder compression process, and Fig. 18 shows the development of stress distribution in a powder bed during the compression formation process.

The stress-strain curve obtained by the simulation coincides closely to measured values, and the simulation therefore provides detailed information on the stress distribution that develops during the compression formation process. In operations for the compression formation of ceramics the particle structure of the formed product must be highly uniform. For that reason it is necessary to limit as much as possible the development of stress distribution in the powder bed during the compression formation process.

This FEM simulation makes it possible to select the optimum operational parameters involved in the compression formation of ceramics, such as powder characteristics, shape of the compression formation vessel, compression rate, compression method, and wall friction coefficient, thereby making it possible to design compression formation operations.

4.2 Powder Behavior Simulation by PEM

The Navier-Stokes equation describes powder flow, but since no fundamental equation had been found for powder flow, it was impossible to simulate it. However, computer advances now make it possible to use the discrete element method, making PEM simulations common, and used also in designing powder processes.

The simulation principle for this method is very simple. Let us assume that the particle group in Fig. 19(a) is flowing in the arrow’s direction. Focusing our attention on one of the particles in the flowing group reveals that its movement is caused by forces imposed by the surrounding particles in contact with it. If it is possible to find the contact force $F$ imposed by surrounding particles from moment to moment, then the movement of the affected particle, which has mass $m$, is given by Newton’s equation of motion $(F=ma)$. Because $F$ and $m$ are known, we can solve the equation of motion and obtain particle acceleration $a$. Integrating the obtained acceleration by time gives the particle’s velocity $v$, and further integration gives its displacement $u$, thereby allowing us to calculate the flowing particle’s trajectory. Because this method gives the trajectories for all the particles involved in fluid movement, it shows the fluid motion behavior of the particle group.

Fig. 20 presents the results obtained when studying the reliability of this simulation method [7].
As shown in Fig. 20(a) we put cylindrical particles in a transparent hopper and discharged them through the bottom orifice. Fig. 20(b) shows the results of recording the cylindrical particles' fluid behavior with a high-speed video camera, while Fig. 20(c) shows the results of simulating fluid behavior using the same conditions as the experiment. These results represent a comparison in which we accurately matched times from the start of movement, and the excellent coincidence demonstrates the great reliability of this simulation. As an example of PEM application, Fig. 21 presents a simulation of particles discharged from a powder hopper. For a long time there have been several puzzles about powder flows in storage hoppers: (1) Wall pressure when the powder bed is stationary in the hopper is not proportional to bed height, but comes to a certain constant value as bed height rises. (2) When opening the orifice at the hopper's bottom and allowing powder to flow out, great pressure is exerted on the hopper wall. (3) The outflow rate and wall pressure vary periodically. (4) The outflow rate is not proportional to the orifice's cross section, but rather proportional to the orifice's diameter to the 2.5-3.0 power. (5) The outflow velocity is constant regardless to the powder bed height.

One advantage of PEM simulations is that, because moment-by-moment information on all flowing particles is calculated, it allows very detailed observations of powder phenomena, which facilitates model analyses of the phenomena. In a study of the aforementioned puzzles about the flow of particles in hoppers, the authors proposed a flow model based on PEM simulation results, and offered an explanation according to dynamics [5].

One more PEM simulation advantage is that it allows one to perform powder experiments in a simple manner for the purpose of process design. Fig. 22 is an example of a screening experiment, and Fig. 22(b) plots the relationship between frequency of the screening apparatus and the residual rate curve. Particle screening is sensitive to frequency, and in this example screening was fastest when frequency $f$ was 14 Hz. In an actual experiment one must conduct tests by preparing a powder sample and making a screening apparatus that conforms to vibration conditions, but computer simulations make it possible to freely vary particle properties and the vibration...
for analyses of powder phenomena and for designing processes. On the other hand, just as with the molecular dynamics method it is difficult to perform large-scale simulations, and it is impossible for the PEM method to directly simulate the behavior of powder in an actual large powder apparatus. In order to get around this drawback, the authors are developing ways to use new simulation methods called the smoothed particle element method and the cellular automaton method [16, 17].

5. Conclusion

This has been an overview of powder simulations for designing powder processes. Especially with more advanced simulations of the powder processing operations included in processes, and with the higher level of production process design, it is necessary to elucidate the relationship between the characteristics of materials produced in processes, and the structure of the materials formed in processes. For that reason we described methods to simulate the relationship between material structure and permittivity, and discussed simulation of sintering, formation, and other processes that form those material structures.

Powder engineering will likely be systematized as a field of engineering pertaining to materials relating to powders and the design of systems for their production processes. It is anticipated that for that purpose powder simulations will become increasingly important. At the same time, the development of computers and improvements in simulation algorithms will proceed at a quickening pace, so that in the near future it will be possible to design powder materials and production process systems with computer simulations.

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^1 Should say which volume.

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Author's short biography

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Prof. Jusuke Hidaka has been at Doshisha University since 1976. His major research interests lie in mechanical analysis of powder flow using computer simulation, classification and instrumentation of powder industrial processes using acoustic emission. He is the secretary of the Society of Powder Technology, Japan.