Three-Dimensional Microstructural Modeling and Homogenization of Porous Alumina with Needle-Like Pores*

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In the development of functional ceramics, the study on the relation between the macroscopic properties and the microstructures has been a critical issue. However, it has not been made clear by many experimental works. On the other hand, this paper takes the computational mechanics approach using the homogenization method. To model the very complex microstructure architecture three-dimensionally, the image-based modeling technique is employed. A porous alumina with needle-like random pores with 3.1% porosity ratio is studied in this paper. The effect of the location of extracting the unit cell and its size on the homogenized elastic constants is investigated. Then, the predicted values were compared with the measured ones. As a result, a unit cell region expressed by one million voxel elements with 2 μm resolution provides very accurate prediction regardless of the location of the unit cell in the random microstructures. The error between the numerical prediction and the measurement is only 1%.

Key Words: Computational Mechanics, Homogenization, Porous Material, Ceramics, Microstructure, Modeling, Numerical Analysis

1. Introduction

Much effort has been devoted to the development of various types of new materials, which aims at saving resources and energy or solving environmental problems. Among them, the synergy ceramics project started in Japan promoted by METI, and many research works have been carried out⁽¹⁾. Synergy ceramics project is a challenge to realize the synergistic effect so that the material has multiple functions simultaneously by controlling many kinds of structural elements ranging from atomic/molecular scale to nano/micro/macro scale as shown in Fig. 1.

Especially, the porous ceramics, which have pores

as the structural element, are becoming a matter of interest because they have many attractive properties such as heat insulation or filtering function used for dust removal etc. These properties are dependent on the microscopic characteristics such as the shape and size of pores and the porosity ratio. Hence, the study on the relation between the macroscopic properties and the microstructures is essential in the design and development of ceramics, and the researchers have long taken experimental and observational approach. However, the relation between the microstructures and macroscopic properties has not been clarified by only the observation of local part or the surface and cross sections of materials two-dimensionally. On the contrary, this study takes the mechanical and computational approach to study the correlation between the microstructures and macroscopic properties for porous ceramics. For this sake, the homogenization method^{(2),(3)} is employed, which is recently studied intensively in the field of composite materials such as fiber reinforced materials. The finite element method is used as a numerical method.

To promote this series of research works, it is indispensable first of all to verify the numerical results

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Fig. 1 Concept of synergy ceramics

obtained by the homogenization method. So far, many numerical examples by this method have been reported. However, we can find only few papers that presented the verification of the numerical accuracy for real materials by comparison with experimental facts. In the field of fiber reinforced plastics, some pioneering works are found^{(4),(5)}. The advantages of the homogenization method are that the macroscopic homogenized properties can be predicted and that the microscopic stress can be calculated based on the microstructure. Concerning the latter point, many difficulties lie in the comparison directly between measurement and numerical analysis by the homogenization method, although new techniques to measure the microscopic stress have been developed such as X-ray⁽⁶⁾, neutron diffraction⁽⁷⁾ and so on. To this end, this paper discusses the comparison of the macroscopic homogenized elastic constants such as Young's modulus and Poisson's ratio between numerical prediction and experimental measurement.

The porous alumina with needle-like pores is studied for the verification of numerical accuracy. Note here that the three-dimensional analysis is required because the pore shape is needle-like and because pores are distributed randomly. Although there have been many reports that studied the randomness⁽⁸⁾⁻⁽¹⁰⁾, no one has ever verified the numerical results by comparison with experimental ones. Since the microstructural modeling is a cumbersome task, one important achievement in the previous works is the image-based modeling technique that was proposed by Hollister and Kikuhci⁽¹¹⁾. This technique is very practical, and therefore is adopted also in our study. Its applications have been reported to the three-dimensional microstructural modeling of metal matrix composites⁽⁸⁾, concretes⁽¹²⁾ and human bones⁽¹³⁾.

Table 1 Measured macroscopic properties

Young's modulus (GPa)	366
Poisson's ratio	0.232

The drawback of adopting the image-based modeling technique is that the large-scale analysis is required using fine voxel mesh. In the authors' previous work⁽¹⁴⁾, therefore, a computer program for homogenization analysis was developed that employs EBE-SCG (Element-by-element Scaled Conjugate Gradient) method as an equation solver. This paper focuses on the image-based modeling of actual porous alumina and on the verification of the numerical results of homogenization using the above-mentioned computer program by comparison with experimental results. Since the properties of ceramics are distributed and the microstructure architecture is random depending on the sintering process, a test specimen is made in this study for the verification of numerical accuracy. Using this specimen, the elastic constants are measured first, and the image-based modeling and numerical analysis have been carried out.

2. Test Specimen

A standard test specimen of porous alumina was produced in this study so that no crack appears during sintering, which can be used for experiment and numerical analysis with variety of pore shape, size and porosity ratios in the series of research work hereafter. Alumina powder and needle-like fugitive constituents mixed uniformly in ethanol were fabricated and sintered. The porosity ratio of the porous alumina used in this paper is 3.1%, which is a measured value after sintering. The average sizes of needle-like pores were approximately 150 μm in length and 10 μm in diameter. A pillar shaped specimen has the diameter of 5 μm and the thickness of 4 μm . In the image-based modeling, the pillar shaped specimen was polished precisely in the thickness direction.

Table 1 shows the measured macroscopic Young's modulus and Poisson's ratio by ultrasonic pulse echo method. These measured values are the properties in the thickness direction of the pillar shaped specimen. At the moment of measurement, isotropy is assumed and the measurement is carried out only in the thickness direction due to the restriction of the dimensions of the specimen. This point is discussed later. Note that the validity of applying the ultrasonic pulse echo method to measure the properties of porous ceramics using the specimen with above-mentioned dimensions has been confirmed by comparison with other measur-

ing methods, as far as the porosity ratio is low such as the above test specimen. The accuracy in measurement and the reproducibility have also been confirmed for the ultrasonic pulse echo method.

3. Image-Based Modeling of Three-Dimensional Microstructure

The test specimen of porous alumina has very complex microstructure. Consequently, the automation of modeling and the development of practical analysis system are indispensable. Hence, the image-based modeling technique⁽⁹⁾ is adopted. This technique enables us to reconstruct the three-dimensional structure as the voxel information from multiple two-dimensional pixel information. To capture the two-dimensional information, such nondestructive method as X-ray CT might be a candidate. However, the resolution of standard X-ray is approximately $5.0\text{ }\mu\text{m}$. That's why X-ray CT can't be used to express the geometry of pores in this specimen with an average diameter of approximately $10\text{ }\mu\text{m}$. To this end, precise polishing and capturing images were repeatedly conducted to acquire the two-dimensional information. We let the resolution $2.0\text{ }\mu\text{m}$ after the authors' previous work⁽¹⁵⁾ that analyzed the correlation between resolution and numerical accuracy for a two-dimensional model with ideally and regularly arrayed circular holes. Also the practicality is considered with respect to the number of repeated polishing and the number of voxel elements.

The schematic of the above procedure and the flow chart are shown in Figs. 2 and 3, respectively. As mentioned before, the pillar shaped specimen of porous alumina is polished precisely by $2.0 \pm 0.25\text{ }\mu\text{m}$ in the thickness direction (denoted as third axis). The cross-sectional image is captured by CCD camera with the resolution of $2.0\text{ }\mu\text{m}$ in the planar directions

Fig. 2 Schematic of image-based modeling

(denoted as 1 - 2 axes). Fifty-one two-dimensional images are obtained after repeating the above procedure. The three-dimensional positioning and the global luminosity adjustment are applied to the obtained two-dimensional information. Next, the alumina matrix phase and the pores are distinguished by binary processing. The voxel mesh is automatically generated using the commercial software VOXELCON (Quint Corp., Japan) from the above data.

Figure 4 shows the reconstructed three-dimensional microstructure of the test specimen by the reverse display of pores. The whole polished region has $800\text{ }\mu\text{m} \times 800\text{ }\mu\text{m} \times 100\text{ }\mu\text{m}$ volume and voxel element size is $2.0\text{ }\mu\text{m}$ according to the resolution of precise polishing. The polished direction is in the

Fig. 3 Flow of image-based modeling

Fig. 4 Distribution of needle-like pores in whole polished region ($800\text{ }\mu\text{m} \times 800\text{ }\mu\text{m} \times 100\text{ }\mu\text{m}$)

third axis indicated in Fig. 4 as mentioned before. Note that, in producing the test specimen, the press forming is carried out in the third axis. The measured macroscopic properties by the ultrasonic pulse echo method are the ones in the third axis.

4. Extraction of Unit Cell and Homogenization

4.1 Outline of homogenization method

The conventional way to model the heterogeneous material was to replace it by the equivalent homogeneous material. The homogenization method^{(2)-(5),(8)-(12)} was developed in 1970s and many researches have been carried out since 1990. Using this method, for arbitrary microstructures, the homogenized macroscopic properties can be predicted and both micro- and macroscopic coupled behaviors can be analyzed. The detail of the theory and formulation of the homogenization method can be found in the published papers^{(1),(2),(14)} and only the outline is summarized in this paper.

We suppose an elastic body defined as Ω in the macroscopic orthogonal coordinate system $\mathbf{x}$. A unit cell Y with its volume $|Y|$ is extracted, which is small region to represent the microstructures spread over the elastic body. We assume that the unit cell is periodically repeated densely. We define the coordinate system $\mathbf{y}$ to describe the microstructure, and then two distinct scales are related by means of the scale ratio ε ($0 < \varepsilon \ll 1$) as $\mathbf{y} = \mathbf{x}/\varepsilon$. Now we suppose that the traction $\mathbf{t}$ is applied to the boundary Γ_t of the elastic body. The equilibrated stress satisfies the following Eq. (1).

$$\begin{aligned} \frac{\partial \sigma_{ij}^\varepsilon}{\partial x_j} + f_i^\varepsilon &= 0 \quad \text{in } \Omega \\ \sigma_{ij}^\varepsilon n_j &= t_i \quad \text{on } \Gamma_t \end{aligned} \quad (1)$$

Here, the upper suffix ε for every variant means that the influence of the microstructure is involved. $\mathbf{f}$ denotes the body force and $\mathbf{n}$ is the normal vector at the boundary.

A displacement field is expanded asymptotically by means of the scale ratio ε as follows.

$$\mathbf{u}^\varepsilon = \mathbf{u}^0(\mathbf{x}, \mathbf{y}) + \varepsilon \mathbf{u}^1(\mathbf{x}, \mathbf{y}) + \varepsilon^2 \mathbf{u}^2(\mathbf{x}, \mathbf{y}) + \cdots \quad (2)$$

Here the second and higher order terms are the displacements due to the heterogeneity involved in the unit cell. In the same way, the stress field can be described by the following asymptotically expanded equation.

$$\boldsymbol{\sigma}^\varepsilon = \varepsilon^{-1} \boldsymbol{\sigma}^{-1}(\mathbf{x}, \mathbf{y}) + \boldsymbol{\sigma}^0(\mathbf{x}, \mathbf{y}) + \varepsilon \boldsymbol{\sigma}^1(\mathbf{x}, \mathbf{y}) + \cdots \quad (3)$$

By summarizing the above relations with respect to the order of ε , we can derive the equations that must be satisfied simultaneously. One important result is expressed by Eq. (4).

$$\boldsymbol{\sigma}^{-1} = \mathbf{0} \Rightarrow \mathbf{u}^0 = \mathbf{u}^0(\mathbf{x}) \quad (4)$$

This means that $\mathbf{u}^0(\mathbf{x})$ is independent on $\mathbf{y}$ and denotes the average displacement when homogenized model of the heterogeneous material is considered. Another important result is expressed by Eq. (5).

$$\text{div}_y \boldsymbol{\sigma}^0 = \mathbf{0} \quad (5)$$

The lower suffix y indicates the coordinate system for differential operation. This is the microscopic equilibrium equation that means that the stress in the unit cell is in self-equilibrium. The volumetric average of this stress in the unit cell is the macroscopic stress as shown in Eq. (6).

$$\langle \boldsymbol{\sigma}^0 \rangle = \frac{1}{|Y|} \int_Y \boldsymbol{\sigma}^0(\mathbf{x}, \mathbf{y}) dY \quad (6)$$

Subsequently, the macroscopic equation that describes the equilibrium against the external force is derived as shown in Eq. (7).

$$\text{div}_x \langle \boldsymbol{\sigma}^0 \rangle + \langle \mathbf{f} \rangle = \mathbf{0} \quad (7)$$

In a word, the homogenization method decouples the governing equation (1) that considers the micro-macro coupling effect into the microscopic equation (5) and macroscopic one (7). Finally, the micro- and macroscopic equations can be solved by finite element method.

4.2 Outline of analysis system

The outline of the analysis system that employs the image-based modeling technique and the homogenization method is shown. The image-based modeling technique is used to generate automatically the three-dimensional model using the voxel (8 node cubic solid) elements from multiple two-dimensional images, a part of which is carried out by the commercial software VOXELCON (Quint Corp., Japan). The generated data is converted to our own analysis program using the homogenization method. The large-scale element subdivision is needed to express the complex microstructures precisely by the voxel elements. To solve the large-scale problem, EBE-SCG (Element-by-element Scaled Conjugate Gradient) method is adopted, which is one of the preconditioned iterative solvers. When 1 to 2 GB core memory is equipped on a standard personal computer (CPU: Pentium III class), problems with up to approximately 2 million elements can be analyzed by this in-core solver. We have already reported the detail of this analysis program using the homogenization method and EBE-SCG method⁽¹⁴⁾. For the visualization, the commercial software Micro AVS (KGT Inc., Japan) is employed considering the portability of the analysis system.

4.3 Influence of location to extract unit cell

In extracting the unit cell that can represent the microscopic information from the test specimen with random microstructure, both objectivity of extraction and computational efficiency must be discussed. The

influence of the location to extract the unit cell on the numerical results is investigated first. Two unit cell models, A and B, are extracted arbitrarily from different location as shown in Fig. 5 in the whole polished region (Fig. 4). Both unit cell models occupy the region of $280\text{ }\mu\text{m} \times 280\text{ }\mu\text{m} \times 100\text{ }\mu\text{m}$ and consist of 980 000 elements. The porosity ratios that are modeled by the unit cells A and B with voxel expression are 3.09% and 3.12%, respectively. These porosity ratios in the unit cell models coincide with the measured value before polishing, which implies that

(a) Location of extracting representative unit cells

(b) Unit cell model A (Porosity 3.09%)

(c) Unit cell model B (Porosity 3.12%)

Fig. 5 Unit cell modeling with 0.98 million elements ($280\text{ }\mu\text{m} \times 280\text{ }\mu\text{m} \times 100\text{ }\mu\text{m}$)

the above models are appropriate with respect to the porosity ratio. In addition, the distribution of needle-like pores seems to be almost homogeneous in the level of the graphical display.

The Young's modulus and Poisson's ratio of the alumina matrix phase used for homogenization calculation are 404 GPa and 0.239, respectively. The obtained homogenized elastic constants are regarded as orthotropic model and are summarized in Table 2. It tells that the predicted homogenized elastic constants by the unit cell models A and B agree quite well within the error of approximately 1.0%. Using the unit cell model that have the resolution of $2\text{ }\mu\text{m}$ to express the microstructure in this test specimen and have approximately one million elements, we can say that it can represent the overall heterogeneity regardless of the location to extract it.

4.4 Influence of unit cell size

Next, two models are studied to investigate the influence of the unit cell size on the numerical results, whose resolution is same with the previous section. The one is a larger model in size (with two million elements) than that in the previous section and the other is a smaller one (with half million elements).

The location to extract the larger model with two million elements ($400\text{ }\mu\text{m} \times 400\text{ }\mu\text{m} \times 100\text{ }\mu\text{m}$) is shown in Fig. 6. This model is located almost at the center of the whole polished region, but is extracted arbitrarily. The porosity ratio expressed by this model is 3.12%, which agrees well with the measured value. The homogenized elastic properties are shown in Table 3. These calculated values by the model with two million elements are in very good agreement with those in Table 2 using a model with one million elements. Note here that the larger model is located at different portion from the previous model. Generally, if the size of the unit cell is enlarged, it is more suited to represent the whole heterogeneity⁽⁹⁾. The above results tell

Table 2 Homogenized elastic constants (0.98 million elements)

Model A	E_{11}, E_{22}, E_{33} (GPa)	379.3, 378.6, 365.3
	G_{23}, G_{31}, G_{12} (GPa)	149.3, 149.5, 153.0
	$\nu_{21}, \nu_{31}, \nu_{32}$	0.236, 0.231, 0.231
Model B	E_{11}, E_{22}, E_{33} (GPa)	381.4, 378.8, 364.0
	G_{23}, G_{31}, G_{12} (GPa)	149.2, 149.9, 153.3
	$\nu_{21}, \nu_{31}, \nu_{32}$	0.236, 0.229, 0.231

Table 3 Homogenized elastic constants (2 million elements)

E_{11} , (GPa)	E_{22} , (GPa)	E_{33} (GPa)	381.3, 377.1, 362.0
G_{23} , (GPa)	G_{31} , (GPa)	G_{12} (GPa)	148.2, 149.2, 153.2
ν_{21} , (GPa)	ν_{31} , (GPa)	ν_{32} (GPa)	0.235, 0.228, 0.231

Fig. 6 Unit cell modeling with two million elements ($400\ \mu\text{m} \times 400\ \mu\text{m} \times 100\ \mu\text{m}$)

us that the unit cell size with approximately one million elements is large enough.

On the other hand, from the viewpoint of computational efficiency, the possibility of using smaller unit cell model is investigated. That is, 16 unit cell models that have half million elements ($200\ \mu\text{m} \times 200\ \mu\text{m} \times 100\ \mu\text{m}$) are extracted as shown in Fig. 7(a). When 16 unit cell models are compared, the porosity ratios vary in wide range from 1.5 to 4.9% (3.1% is the measured value) and also the calculated homogenized elastic properties vary. Among them, the unit cell models No. 5 and No. 15 have relatively closer porosity ratios to the measured value, which are 2.92% and 3.39%, respectively.

The homogenized elastic properties obtained by these unit cell models are shown in Table 4. No

(a) Location of extracting unit cells

(b) Unit cell model No. 5 (Porosity 2.92%)

(c) Unit cell model No. 15 (Porosity 3.39%)

Fig. 7 Unit cell modeling with 0.5 million elements ($200\ \mu\text{m} \times 200\ \mu\text{m} \times 100\ \mu\text{m}$)

remarkable difference between Tables 2 and 3 is found. However, the smaller model with half million elements is lacking in objectivity because the location of extracting unit cell model is very much influential on the numerical results. It is concluded from the above results that the unit cell model in section 4.3

Table 4 Homogenized elastic constants (0.5 million elements)

No.5	E_{11}, E_{22}, E_{33} (GPa)	380.1, 380.2, 368.7
	G_{23}, G_{31}, G_{12} (GPa)	150.6, 150.1, 153.5
	$\nu_{21}, \nu_{31}, \nu_{32}$	0.237, 0.230, 0.232
No.15	E_{11}, E_{22}, E_{33} (GPa)	379.4, 377.0, 359.7
	G_{23}, G_{31}, G_{12} (GPa)	148.1, 148.6, 152.6
	$\nu_{21}, \nu_{31}, \nu_{32}$	0.236, 0.228, 0.230

with approximately one million elements ($280\ \mu\text{m} \times 280\ \mu\text{m} \times 100\ \mu\text{m}$) is best from viewpoints of computational efficiency and objectivity. The size of the voxel element is $2.0\ \mu\text{m}$. When we analyze the material whose heterogeneous microstructure has the same representative dimension with the test specimen in this paper, the above modeling strategy to determine the unit cell size is supposed to be applicable regardless of its location.

4.5 Discussion

In the above sections, based on the numerical results only, we found the objective modeling strategy to extract the unit cell. Here we add some discussion on the convergence of EBE-SCG iterative solver for the microscopic equation in the homogenization method from the standpoint of the computational efficiency. Figure 8 shows an example of convergence when the unit cell model A with one million elements is analyzed. The horizontal axis denotes the number of iterations and the vertical one denotes the relative residual error, which is defined by the norm of the residual error vector in the CG iteration divided by the norm of the right hand side vector of the linear algebraic equation.

Figure 8 tells that the convergence check is whether the relative residual error is less than 10^{-3} . Note here that the calculated homogenized elastic constants have no remarkable difference when we let the threshold be 10^{-3} to 10^{-6} . Also note that the iterative solver works quite well for this kind of analysis.

Finally, the numerically predicted results are compared with measured values. Note here that the calculated elastic constants show isotropy in 1-2 plane but show anisotropy in the third axis by approximately 5%. Carefully looking at the three-dimensional microstructure reconstructed by the image-based modeling, we can find that the longitudinal

Fig. 8 Typical example of convergence in EBE-SCG iteration

direction of needle-like pores is oriented in the 1-2 plane. It is reasonable that this orientation results in lower Young's modulus in the third axis as predicted. It is supposed that the forming process in fabricating the test specimen reoriented the fugitive needle-like inclusion. We could not predict this phenomenon that leads to the anisotropic property at the time of measurement, and consequently we have only one measured value in the third axis as shown in Table 1. Comparing the Young's modulus in Table 1 with one in the third axis E_{33} in Tables 2 and 3, the error is approximately 1% and both results agree quite well. Same is true for the Poisson's ratio.

In conclusion, the above-presented strategy to extract the unit cell provides accurate prediction of the macroscopic properties independently on the individual analyst. Another result to support this conclusion is that the numerical analyses of models with coarse resolution generated from the image-based model with $2\ \mu\text{m}$ resolution were very sensitive to the strategy to extract the unit cell. Therefore, at this moment, a non-destructive method to obtain the images is not suited to model the porous material whose representative dimension of pore is approximately $10\ \mu\text{m}$.

5. Concluding Remarks

The obtained results in this research are summarized as follows.

(1) To predict the macroscopic properties of porous ceramics in consideration of their complex microstructures, the practical analysis system has been developed using the image-based modeling and homogenization method. This system is capable of analyzing large-scale problems with up to two million elements on a standard personal computer.

(2) The influence of the location to extract the unit cell and its size on the predicted macroscopic

properties has been investigated for a standard porous alumina specimen with needle-like pores whose average diameter and length are 10 μm and 150 μm , respectively. It has been found that 2.0 μm resolution, i.e., the size of a voxel element, is needed to express the microstructure and that the strategy to extract the unit cell with region subdivided by approximately one million elements provides objective numerical results independently on the individual analyst.

(3) It has been confirmed that the numerically predicted properties agree quite well with the measured ones within approximately 1% error.

The application of the developed system to other materials with different shape and size of pores and different porosity ratios is now ongoing. That is, the same numerical analysis and experiment with this paper are carried out, for instance, for porous alumina specimens that have spherical pores with approximately 20 μm average diameter and porosity ratio of below 4%. Again we have nice results similar to this paper⁽¹⁶⁾. Another specimen under construction may have controlled anisotropy. Those new results are to be appeared in our next report.

One remark to be mentioned is that very accurate numerical prediction has been successfully performed in this paper because identical specimen has been used for both image-based modeling and measurement. Since the porous ceramics have variation in nature, one future work should be devoted to propose a general-purpose strategy for their microstructural modeling.

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