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Electro-thermo-mechanical finite element analysis on DC pulse resistance pressure sintering process of zirconia part

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Abstract

Some difficulties have arisen on appropriate design of the graphite dies, on applying the DC pulse resistance pressure sintering process, which is more efficient process than other conventional sintering processes such as hot pressing process, to a complex shaped part of non-conductive ceramics powder such as zirconia powder. In the present paper, the non-steady electro-thermo-mechanical finite element analysis of the sintering process of the zirconia cylindrical can was performed. As a result, it was found that the enough heating of the graphite dies that contacts with zirconia powder, and heat transmission from the die to zirconia powder was essential for the success of the sintering with adequate density. Moreover, the lower density region was found at the inside of the can during the process, which corresponded to the region where the convex shape formed in experiments. Furthermore, the high tensile residual circumferential stress was observed at the side wall from the simulation of the cooling process, which is caused by the different thermal expansion ratio of the graphite and zirconia materials. This stress was considered to be related with the fracture of the can longitudinally during cooling process in experiments.

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1. Introduction

DC pulse resistance pressure sintering process, called as the SPS process, is an efficient sintering process (Inoue, 1963). Sintered parts can be made in short time by the combination of resistance heating and pressure. For the complex shape of electrically non-conductive ceramics powder such as zirconia powder by the process, there have been some difficulties on designing the graphite dies and tools, because of unclear information of the distribution of electrical current, temperature, relative density, and the resulting mechanical property of sintered part. Especially, the information of the heat transmission behavior from die to the ceramic power is very important, because the electrically non-conductive ceramic powder cannot generate heat by resistive heating. Numerical analysis can help the development of proper sintering process design of the net shaped dense part of zirconia with complex geometry.

There are some researches on electro-thermo-mechanical coupled FE analyses for the process of simple disk-shaped part (Wang et al., 2007; Wang et al., 2010; Bellet et al., 2010). The present author performed the process analysis of titanium thin-walled can part (Kubota et al, 2012). They also performed the process analysis of zirconia can-shaped part (Tagashira et al, 2012). In those analyses the rigid-plastic deformation was assumed for the deformation of titanium and zirconia, and the deformation of graphite dies was neglected. For the process of zirconia can-shaped part, the consideration of the residual stress during the process is very important for the evaluation of fracture after completion and ejection, since zirconia is rather brittle.

In the present paper, the non-steady electro-thermo-mechanical finite element analysis of the DC pulse resistance pressure sintering process of the zirconia cylindrical can-shaped part was performed for the establishment of the proper sintering process design. For mechanical calculation, elastic-plastic property of the zirconia and elastic property of graphite is employed for the evaluation of the residual stress generated in both materials.

2. FEM simulation on sintering process of zirconia disk

2.1. Finite element modeling

The sintering process for zirconia disk was first analysed. Boundary conditions and material behaviour by compared with the calculation and the experiment. Fig. 1(a) shows the calculated model consisting of die, upper graphite punch and lower graphite punch and zirconia powder. The zirconia powder filled in the die cavity is compressed by the lower graphite punch at a constant load of \( P = 4.72 \text{ kN} \). Subsequently, the zirconia powder was sintered by the heat conduction from the graphite die heated by pulsed electric current as shown in Fig. 1(b). Temperature was measured at the measurement point using thermocouple. The prescribed dimensions of the disk was \( d = 10 \text{ mm} \) in diameter and \( h = 5 \text{ mm} \) in height.

![Fig. 1. Analysed model for zirconia disk part sintering process and conditions of compressive load and applied voltage: (a) Graphite dies for zirconia disk and (b) Change of applied voltage and compressive load for the sintering process of zirconia disk.](image_url)
The graphite material was modelled as elastic body. The zirconia powder was modelled as a compressible elastic-plastic continuum material with large deformation. The yield function $F$ was given as a function of von-Mises equivalent stress, hydrostatic stress and relative density of material $\rho_R$ proposed by Shima-Oyane as shown in Eq. (1) (Shima et al., 1976, 1986). The $\gamma$ and the $\beta$ is a function of $\rho_R$. These were determined by comparing the calculated with experimental results as shown in Eq. (1).

$$F = \frac{1}{\gamma} \sqrt{\frac{3}{2} \sigma^\prime : \sigma^\prime + \left(\frac{\sigma_m}{\beta}\right)^2} - \sigma_y, \quad \gamma = \rho_R^{5.50}, \quad \beta = \frac{0.40}{\sqrt{1 - \rho_R}},$$

where $\sigma^\prime$, $\sigma_m$ and $\sigma_y$ are the deviatoric strain tensor and mean normal stress and yield stress of zirconia. $\sigma_y$ is assumed as temperature dependent. The associate flow rule was assumed for the derivation of plastic strain rate.

Figures 2 shows the changes in stroke of lower punch $S$ and temperature at measurement point in the die $T$. The calculated $S$ and $T$ is also plotted in these figures. From the experiment, the $S$ progressed rapidly after $t = 300s$, at which the $T$ was around $850^\circ$C. From the result, the $\sigma_y$ can be given as a function of $T$ as shown in Fig. 3(a).

![Fig. 2](image1)

**Fig. 2.** Comparison with experiment and analysis for change of punch stroke and temperature during sintering: (a) Change of punch stroke and (b) change of temperature at measurement point in Fig. 1.

![Fig. 3](image2)

**Fig. 3.** Mechanical properties of zirconia powder during sintering: (a) Relationship between yield stress and temperature and (b) relationship between young modulus and relative density.

![Fig. 4](image3)

**Fig. 4.** Electric and thermal properties of graphite (a) electric resistivity (b) Heat-transfer coefficient of graphite.
The \( y \) and the \( \beta \) in Eq. (1) were determined so that they can express the sintering behaviour of zirconia disk properly. Young’s modulus of the zirconia \( E_z \) was given as a function of relative density \( \rho_R \) as shown in Fig. 3(b).

The electric and thermal properties on the graphite were assumed to be dependent on temperature as shown in Fig. 4. The boundary conditions on the compressive load \( P \) and the applied voltage \( V \) to the die and tools during the sintering were given as same as the experimental ones performed by the present authors as shown in Fig. 1(b).

The temperature of surfaces of the upper and lower electrode was assumed to be 20\(^\circ\)C constantly due to water-cooled. For the other physical and mechanical properties, the values in Table 1 were used. The FE code Marc 2012 (MSC.Software) was used for the calculation. In the present analysis the sintering stress was not considered, since the effect of the sintering stress is less than that of the stress by compression in the present process.

### Table 1. Physical and mechanical property of graphite and zirconia.

| Physical and mechanical properties | Graphite | Zirconia |
|-----------------------------------|----------|----------|
| Density \( \rho / \text{kg} \cdot \text{m}^2 \) | 1850 | 6000 |
| Young's modulus \( E / \text{MPa} \) | 13700 | Fig.3(b) |
| Poisson's ratio \( \nu \) | 0.2 | 0.3 |
| Specific heat \( c / \text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1} \) | 710 | 502 |
| Heat-transfer coefficient \( h / \text{W} \cdot \text{m}^{-2} \cdot \text{K}^{-1} \) | Fig.4(b) | 3.00 |
| Electric resistivity \( \rho_e / \Omega \cdot \text{m} \) | Fig.4(a) | \( 1.00 \times 10^{7} \) |
| Thermal expansion ratio \( \alpha / \text{K}^{-1} \) | \( 5.50 \times 10^{-6} \) | \( 10.0 \times 10^{-6} \) |

#### 2.2. Calculated results

The calculated \( S \) and \( T \) shown in Fig. 2 are in good accordance with the experimental results. Fig. 5 shows the calculated distribution of \( T \) and \( \rho_R \) at \( t = 720 \) s when the sintering is finished. The \( \rho_R \) is almost unity in whole region without outside upper and bottom edge. In experiment, only \( T \) at the measurement point (Fig. 1(a)) can be observed. When the \( T \) at the measurement point exceeds 1300 \( ^\circ\)C, the \( T \) in the part is over 1290 \( ^\circ\)C and \( \sigma_m \) is almost around 60 MPa in the whole region at the completion of sintering, which corresponded with the experimental ones by the present authors.

#### 3. FEM process simulation on sintering of zirconia cylindrical can-shaped part

Figure 6 shows the model of the graphite die and tools for sintering zirconia cylindrical can-shaped part. The prescribed dimensions of the part is \( d = 10 \) mm in diameter, \( t = 1 \) mm in thickness of the bottom and wall at the top, \( \alpha = 8^\circ \) in tapered angle of the wall, which was proposed by the present authors (Tagashira et al., 2012). This construction can be made to enough loads the average normal stress \( \sigma_m \) needed in the wall and bottom of can. These are composed with electrode, distance piece, upper punch, lower punch and die. The zirconia powder is compressed between the lower punch and upper punch as the lower punch goes up. The sliding length of upper punch \( L = 18.22 \) mm can be calculated by taking the initial relative density \( \rho_{RI} = 0.23 \) of the zirconia. The applied
voltages and compressed load were shown in Fig. 6(b). The initial conditions of the calculation are the same as the table 1 in the previous section.

Figure 7 shows change in $T$ at the measurement point (Fig. 6(a)), the distribution of $T$ and $\rho_R$ at $t = 420$ s; completion of sintering. From the calculated results, it was found that the enough heating of the graphite dies that contacts with zirconia, and heat transmission from the die to zirconia was essential for the success of the sintering with adequate density. The $\rho_R$ was over $\rho_R = 0.98$ in whole region except for the inside wall.

The region of lower $\rho_R$ was also found at the inside of the can during the deformation process as shown in Fig. 8(a), which corresponded to the region where the convex shape formed in experiments.

Furthermore, the large tensile residual circumferential stress was observed at the side wall from the calculated result of the cooling process as shown in Fig. 9. Though the compressive circumferential residual stress was observed at the start of cooling process, $t_c = 0$, the high tensile circumferential stress was considered during cooling process $t_c = 120$ s and 360s. This circumferential stress was considered to be related with the fracture of the can in longitudinal direction after the removal of the can from the graphite dies in experiments. The high tensile circumferential stress came from the different thermal expansion ratio of the graphite and zirconia materials.
5. Conclusion

Electro-thermo-mechanical coupled FE analysis of the DC pulse resistance pressure sintering process of zirconia can-shaped part was performed to evaluate the temperature, relative density distributions and residual stress at the cooling process.

From the analytical result, the electro-thermo-mechanical coupled FE analysis was found to be useful for proper design of dies and process. Moreover, lower relative density region was obtained inside of wall by analysis, where the convex stepped shape was observed by the experiment. Finally, the residual tensile circumferential stress during cooling process was found to be main cause of longitudinal fracture of zirconia can. Such a high circumferential stress was caused by the different thermal expansion ratio of the graphite and zirconia materials.

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