IN-SITU STRENGTH OF ALUMINA GREEN COMPACT AT ELEVATED TEMPERATURE DURING SINTERING

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ABSTRACT

Recently, ceramics is extensively applied to structural materials and ceramics components become larger and more complex. Because of its large and complex shape, ceramic components are often prone to fracture in the sintering process. The simulation of sintering has been investigated to prevent the fracture. However, simulation of ceramics sintering was difficult because the strength of the ceramics during sintering has not been well investigated so far. In this study, we measured strength of alumina green compact during sintering and proposed models for strength. Two kinds of alumina green compact were used to measure strength. Three point bending test at elevated temperature was performed and strength was estimated at each temperature. Three models based on creep behavior of sintered ceramics were also proposed. The strength was evaluated by deformation stress because of the ductile manner above 1373K. The strength for high strain rate increased until about 1573K and decreased over 1573K. In contrast, the strength for low strain rate decreased with temperature. The effect of density and strain rate on strength was also investigated. During low shrinkage rate, there was no effect of relative density on strength. The strength increased with strain rate, so the effect of strain rate was also observed. Furthermore, the experimental results were compared to models for strength. Two models were in good agreement with the experimental results. Accordingly, the strength was predicted by these models and experimental parameters. We could predict the strength and the effect of temperature and strain rate on strength at elevated temperature. This result permits us to carry out the simulation of firing and we expect these models applies another ceramics materials.

1 INTRODUCTION

Ceramics is used for structural materials because of its strength, abrasion resistance, chemical stability and so on. Ceramic components are produced by heating and densifing green compact at elevated temperature. These components are sometimes easily fractured during sintering due to large size and complex shape of ceramic components, so it becomes a problem that this fracture interrupts manufacturing process. It is important to control the crack initiation and propagation in green compact during sintering to resolve this problem. Furthermore, it is necessary to understand the evolution of mechanical properties during sintering to control the crack behavior.

The firing process is divided into four stages such as defatting, heating, sintering and cooling process. The temperature distribution exists because of the heat transfer from surface during firing. Internal stress is induced by the pyrolysis gas at defatting process, the nonuniformity of shrinkage at sintering process and the temperature distribution at heating and cooling process (e.g. Harato [1]). It is necessary to keep the induced stress below the fracture strength during firing in order to prevent a fracture and obtain a sound sintered body.

The simulation of the sintering has been studied to prevent the generation of fracture and deformation. Miyata et al. (e.g. Miyata [2], [3], [4]) simulated the stress distribution during sintering using the equation of shrinkage rate. Mechanical properties of green compact during sintering are very important to carry out this simulation. However, it was difficult to simulate fracture because there is almost no report on mechanical properties. In this paper, we measured strength during sintering at elevated temperature and predicted it by using models for strength.

2 ANALYTICAL PROCEDURES

It is difficult to exactly evaluate the strength of green compact during sintering because materials state always changes with time. In present study, we tried to simplify this problem and represent the strength as a function of testing temperature. Generally, strength of materials was represented as a function of materials state and testing condition, so we assumed the strength during sintering was also represented as a function of these parameters. Accordingly, we should investigate the effect of these parameters such as density, testing temperature and strain rate on the strength during sintering.

Ceramics materials demonstrated ductile manner and creep deformation at elevated temperature. Three models related to creep mechanism were examined to estimate the strength during sintering. Firstly, we used the general creep behavior as a model for strength, and the relationship between stress and strain during creep deformation in the sintered materials was given by

$$\sigma = K \cdot \dot{\varepsilon}^N \cdot \rho^M \cdot T \cdot \exp\left(\frac{Q_0}{RT}\right) \qquad (1)$$

where σ is stress, $\dot{\varepsilon}$ is strain rate, ρ is density, Q_0 is activation energy of sintered ceramics and K, N and M are material parameters, respectively. This model is denoted by model 1. Then, we proposed model 2 where activation energy during sintering, Q, was substituted for the activation energy, Q_0, as follows,

$$\sigma = K \cdot \dot{\varepsilon}^N \cdot \rho^M \cdot T \cdot \exp\left(\frac{Q}{RT}\right) \qquad (2)$$

In the previous study (e.g. Harato [1]), the model which included the effect of grain growth during sintering was suggested, so we used this model as model 3, as follows,

$$\sigma = K \cdot \dot{\varepsilon}^N \cdot \rho^M \cdot \exp\left\{-\left(l_1 T + l_2 T^2\right)\right\} \qquad (3)$$

where l_1 and l_2 are experimental parameters. The strength during sintering was analyzed by these three models.

3 EXPERIMENTAL PROCEDURE

3.1 Materials

The materials in present study consisted of two kinds of alumina (99.5% purity), A115 and A112, respectively. Alumina green compact was obtained by uniaxial compression (14MPa, 10min) and cold isostatic press (140MPa, 5min), and the average density of both green compacts was about 2.3g/cm^3. Microstructure by SEM observation showed A115 had large grain compared with A112 after fully sintered. The shrinkage rate of A115 and A112 was also measured, and this result demonstrated that A112 was easily densified compared with A115. Each green compact was defatted before bending test.

3.2 Three point bending test at elevated temperature

Bending test was performed to estimate strength during sintering. Alumina green compact was cut to the three point bending specimen, 30 by 6 by 4 mm. Each test was started immediately when temperature reached the testing temperature. The heating rate was 5.56x10^{-2}K/s until 1373K, and 1.39x10^{-2}K/s over 1373K. Cross head speed (CHS) was selected as 0.833x10^{-4}, 0.167x10^{-4}, 0.833x10^{-5}m/s to investigate the effect of strain rate.

4 RESULTS

Brittle behavior was observed in the load-displacement curve at low temperature and high CHS, while ductile behavior was observed at high temperature and low CHS. The strength was estimated from rapture stress in case of the brittle behavior and deformation stress in case of the ductile behavior. The effect of testing temperature on strength of each alumina was shown in Figures 1 and 2. Opened marks and closed marks indicate ductile and brittle behavior during tests, respectively. Strength was corrected by the change of geometry due to shrinkage at each temperature which was estimated by shrinkage curve, and then the effect of density on strength is shown in Figures 3 and 4. The effect of strain rate on strength was also investigated because materials during sintering demonstrated ductile behavior like creep deformation. In this study, strain rate was estimated by stress rate and Young’s modulus. This result showed the effect of strain rate on strength.

In the temperature range where specimens showed ductile behavior, we compared experimental results with fitted results by our models. Although model 1 could not fit the experimental results adequately, fitted results by model 2 and 3 were in good agreement with the experimental results. The comparison between fitted results by model 3 and experimental results for A115 and A112 are shown in Figures 5 and 6, respectively. It was demonstrated that the strength of alumina with plastic deformation could be predicted by using these models. The simulated strength of A115 and A112 by model 3 using these experimental results is shown in Figures 7 and 8, where the strength with the effect of strain rate can be predicted in the ductile deformation.

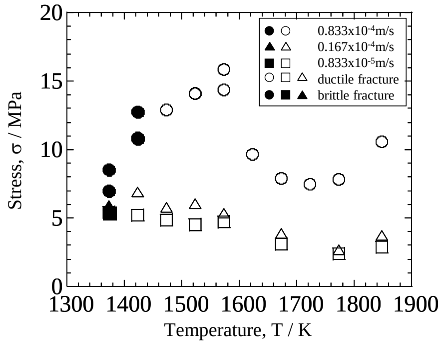

Figure 1: Strength of A115 during sintering.

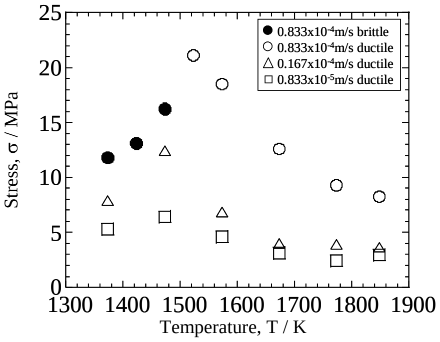

Figure 2: Strength of A112 during sintering.

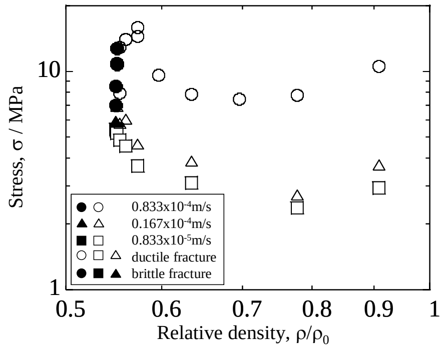

Figure 3: Relationship between stress and relative density of A115.

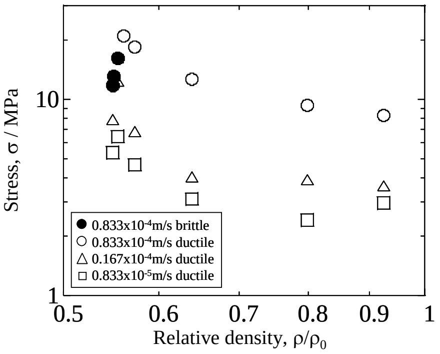

Figure 4: Relationship between stress and relative density of A112.

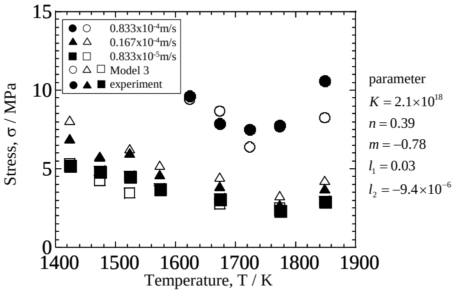

Figure 5: Comparison between model 3 and experimental results for A115.

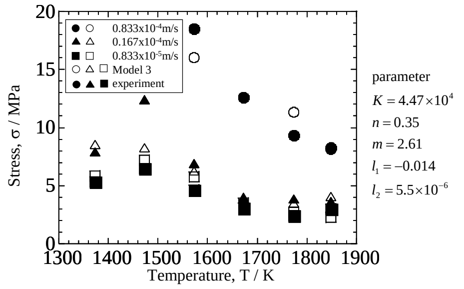

Figure 6: Comparison between model 3 and experimental results for A112.

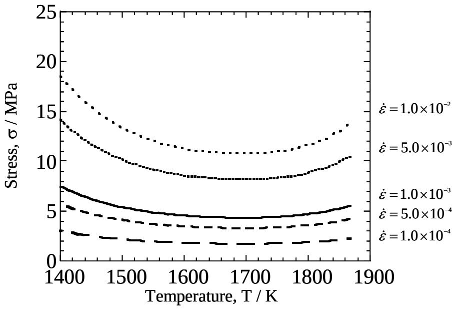

Figure 7: Calculation of strength with the effect of strain rate for A115 by model 3.

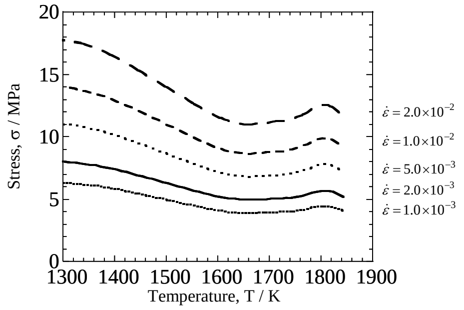

Figure 8: Calculation of strength with the effect of strain rate for A112 by model 3.

5 DISCUSSION

The shape of load-displacement curve implied that the strength in ductile behavior was affected by creep deformation. In Figures 3 and 4, it was difficult that strength in brittle behavior was represented as a function of relative density, because strength increased in spite of a little shrinkage rate. Accordingly, the strength in this range was mainly dependent on the increase of Young’s modulus, not on the creep behavior. In contrast, there was the effect of relative density on strength in ductile behavior. The effect of strain rate on strength in ductile behavior was also observed, so the strength in ductile behavior was predicted by the model based on creep deformation.

Three models for strength of ceramics for the ductile behavior at elevated temperature were examined. Model 1 for strength prediction was based on the effect of creep deformation of sintered ceramics. However, the fitted results by model 1 were not in agreement with experimental results. This disagreement showed that the mechanism of strength during sintering was different from the creep mechanism of sintered ceramics. In model 2, the activation energy of sintered ceramics was substituted for that of sintering ceramics. Consequently, there was good agreement between fitted results by model 2 and experimental results. The activation energy is an important parameter of sintering. However, the activation energy during sintering was not evaluated easily in case of existing impurities in green compact (e.g. Fang [5]). Fang et al. (e.g. Fang [6]) suggested the equation affected by the parameters of size distribution and morphology of pore. Because these parameters were sensitive to testing temperature and heating rate, the activation energy was also influenced. If these parameters were represented as a function of processing conditions, the activation energy could be evaluated. Model 3 was based on the experimental equation, so parameters in model were obtained from the regression constants by experimental fitting. Figures 7 and 8 show that the effect of testing temperature and strain rate on strength is simulated using this model 3. Consequently, these results permit us to carry out the simulation of firing. Furthermore, it is expected that strength of another ceramics during sintering is evaluated by this model.

6 CONCLUSIONS

In present study, the strength during sintering was measured and models for strength were examined to carry out the simulation during firing. The following conclusions could be obtained.

(1) Three point bending test at elevated temperature was performed. Brittle behavior was observed at low temperature and high CHS, in contrast ductile behavior was observed at high temperature and low CHS.

(2) Strength during sintering was depended on strain rate and relative density, and mainly dominated by creep deformation. Strength could be represented as an experimental equation including strain rate, relative density and temperature.

(3) Strength during sintering could be simulated by obtained experimental parameters. This result can permit us to calculate stress distribution and predict a fracture during firing.

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