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Microwave sintering of Fly Ash substituted body

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석탄회가 첨가된 점토의 마이크로파를 이용한 소결

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Abstract Fly ashes mixed with clay as 70:30 weight percent were sintered by microwave energy and a 2.45 Ghz kitchen model microwave oven was used. Samples were sintered at 1,150℃ and kept at that temperature up to 50 minutes by 10 minutes intervals. Microstructures were taken by Scanning Electron Microscope (SEM) and Energy Dispersive Spectrometry (EDS) analysis of a raw fly ash was taken. X-ray diffraction analysis was done, and compressive strengths and apparent densities were measured. Pore sizes of the samples became smaller as time passed by, but compressive strengths and apparent densities did not change much. Numerical analysis on the microwave heated system was carried out in order to figure out heat transfer phenomena in the cavity.

요 약 석탄회와 점토를 70:30의 비율로 혼합하여 마이크로파를 이용하여 소결하였으며 소결에는 2.45 Ghz의 일반 가정용 마이크로파 오븐을 사용하였다. 시편은 1,150℃에서 50분까지 10분 간격으로 소결하였다. 석탄회의 미세구조는 주사전자현미경(SEM)과 Energy dispersive spectrometry(EDS)를 이용하여 분석을 하였으며 X선회절 분석과 압축강도와 겉보기 비중을 추정하여 비교하였으며 시간이 지날수록 시편의 기공크기는 작아졌으나 압축강도와 겉보기 비중은 변화의 크기가 작았다. 로안에서의 마이크로파에 의한 시편으로의 열전달현상을 알아보기 위하여 컴퓨터를 이용한 수치해석을 행하였다.

1. Introduction

Fly ash is a huge amount of waste product of the coal-burning electric power plant. The amount of fly ash was around 3 million tons in 1996 and expected to be more than 7 million tons in 2,000, domestically. The recycling rate in Korea is around 10 percent while it is around 30~50 percent in advanced nations [1].

Microwave processing is one of new processing methods in ceramic materials and has advantages such as fast firing, selective heating, heating from inside of a sample and cost savings, etc. [2, 3].

The sample was heated with SiC as a suscepting material by using a microwave hybrid heating (MHH) method since most oxide ceramic materials absorb microwave energy a little at low temperature. This SiC susceptor absorbs the microwave energy and heats up very fast. The sample itself absorbs the microwave energy and converts it to heat effectively after the temperature passes the critical temperature (Tc). The effective dielectric loss factor of the sample becomes high as the temperature goes up by the radiating heat from the susceptor.

In this study, Boryong fly ash mixed with clay as a 70:30 weight percent ratio was sintered by using microwave energy. Computer simulation was done to investigate the heat transfer phenomena between the susceptor and the sample in the mold.

2. Numerical Analysis

The system for the numerical analysis consisted of a disk-type fly ash substituted body in a cylindrical SiC susceptor which is boxed in an

Isolite insulating cubic container (1.72×10^{-3} m^3), and then, enclosed in a large cavity as in Fig. 1. It was assumed in the calculation that the microwave field is uniform over the whole surface of the system being heated. The field strength within the insulation and the substituted body was calculated as described below. Though the insulating container was cubic, it was modeled as a cylindrical coordinate system according to the body and the susceptor. Thus, the problem was formulated as a 2-dimensional (r-z) cylindrical coordinate system. The basic mathematical formulation can be stated [4, 5] as

$$\frac{1}{r}\frac{\partial}{\partial r}\left(kr\frac{\partial T}{\partial r}\right)+\frac{\partial}{\partial z}\left(k\frac{\partial T}{\partial z}\right)+q(r,T)=\rho C_p\frac{\partial T}{\partial t} \quad (1)$$

where, T represents the absolute temperature at location r at time t, ρ is the density of the material, C_p is the heat capacity at constant pressure, k is the thermal conductivity, and q(r,T) is the volumetric heat generation rate created by dissipation of microwave energy, q is computed from

$$q(r,T)=2\pi f\varepsilon_0\varepsilon_r'\tan\delta E^2 \quad (2)$$

where f is the frequency, ε_0 is the permeability of vacuum, ε_r' is the relative dielectric constant, tan δ is the loss factor, E is the electric field in the sample.

Eq. (1) describes the temperature in both the sample and the insulating container and as well as cavity, when appropriate properties are used for each. As stated above, the microwave field strength E_f is assumed uniform over the whole surface of the container. Within the container, E_f is decreased by the reflection at each interface, and is attenuated exponentially with distance from the surface using the skin depth approach.

In the calculation, the critical temperature was taken as 500℃, approximately, which was decided from the experiment. Below 500℃ heat flux generated at the SiC susceptor was calculated as 1.7 kW/m^3 and it was 2.2 kW/m^3 for the susceptor temperature over 500℃.

The equation was solved using a fully implicit finite difference scheme. Total 50×56 meshes were used in this calculation. The time stepsize started at 1 sec and managed to be decreased according to convergence-divergence characteristics [6].

The most important thing in the mathematical modelling is to transfer precisely the physical concept into the equation. In most cases, material properties were treated as temperature-dependent, where reasonably reliable data were available.

Based on the preliminary experiments, it was found that the density of the body varied a little, and thus density changes were ignored in these calculations. Even in case of changes in density, it might have little differences in the temperature distribution.

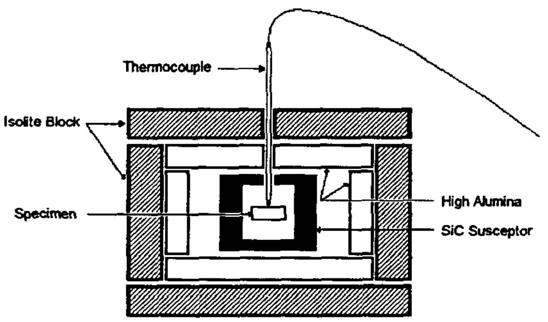

Fig. 1. A schematic diagram of the microwave heating system.

3. Experimental Procedures

Boryong fly ash was used in this study and its chemical composition is listed in Table 1. The fly ash was mixed with clay as a 30:70 weight percent ratio and the detailed procedure to make the greenbody was published in reference 1. The density of the slurry to make the greenbody was 1.6.

A simple 2.45 Ghz kitchen model microwave oven (LG Electronics; Model MR-M274, 750 W) was used in this experiment and a schematic diagram of a mold setting is shown Fig. 2. Temperature was

Table 1
Chemical compositions of Clay and Boryong Fly Ash

	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	TiO_2	P_2O_5	C	Ig-loss
Clay	61.67	22.74	3.49	0.61	0.48	0.42	1.57	0.29	0.05	-	8.67
Fly Ash	65.28	22.87	3.93	0.85	0.55	0.24	0.91	1.22	0.25	-	3.82

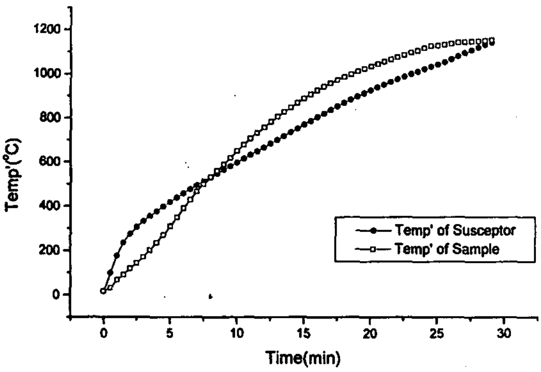

Fig. 2. Temperature profile of the sample and the susceptor.

increased by 40°C/min up to 1,150°C which was decided from the previous experiment with a conventional electric furnace and kept at that temperature from 10 minutes to 50 minutes by 10 minute intervals. Sintered samples were labelled from S1 to S5 according to the time intervals.

Microstructures of the sintered samples were taken by Scanning Electron Microscope (Topcon SX-30E) with an Energy Dispersive Spectrometry (EDS; Kevex Superdry 3866-0698-0149). Fly ash raw materials were imbedded in epoxy and cut by diamond saw to see the compositional distribution within a fly ash particle by EDS and bulk densities of the sintered samples were measured by an Archimedes method. 2 pieces of 5 mm×5 mm×10 mm size rectangular shaped bar were cut out of the each sintered sample and compression tests were done with a press.

Temperature increments of the suscepor and the sample were measured simultaneously to find out the critical temperature (Tc) up to 1,150°C and is shown in Fig. 2.

4. Results and Discussion

4.1. Property analysis

SEM micrographs of sintered fly ash is shown in Fig. 3. Necking had been progressed in the sample as time passed and the micrograph of S5 shows smaller sized pores than those of S1 in Fig. 3. But the compressive strength was 1,200 Kg/cm^2

The bulk densities of the sintered samples showed a slight increment with time passed by and they are shown in Fig. 4.

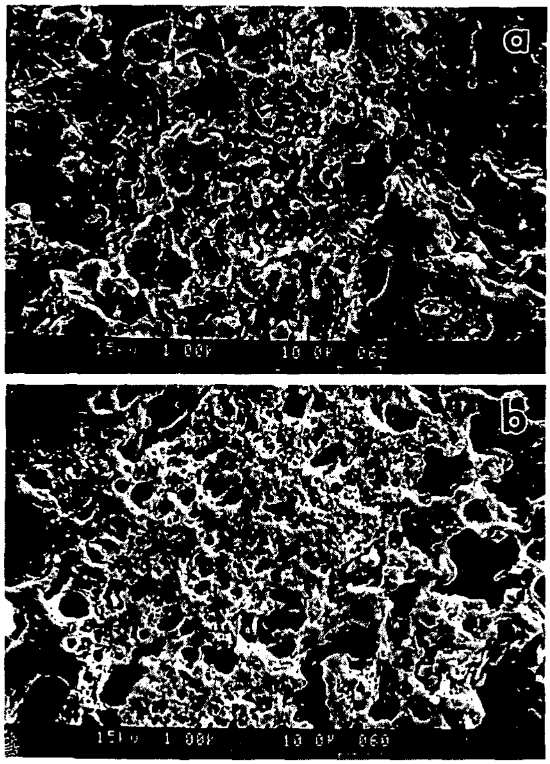

Fig. 3. SEM micrographs of sintered samples, a) after 10 min. b) after 50 min.

Compositional distribution is one of the important factors which affect sintering characteristics if compositions with low melting point exist in the near surface area. These compositions could form a protective layer. Hence a single fly ash particle was mapped by EDS as in Fig. 5. Compositional distribution of Fe was homogeneous and others were concentrated in the near surface. Because the

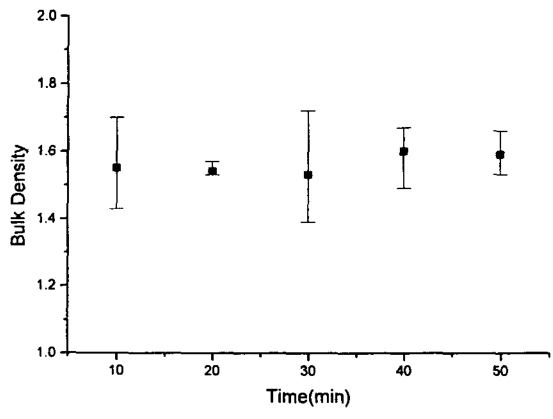

Fig. 4. Bulk densities at each time intervals.

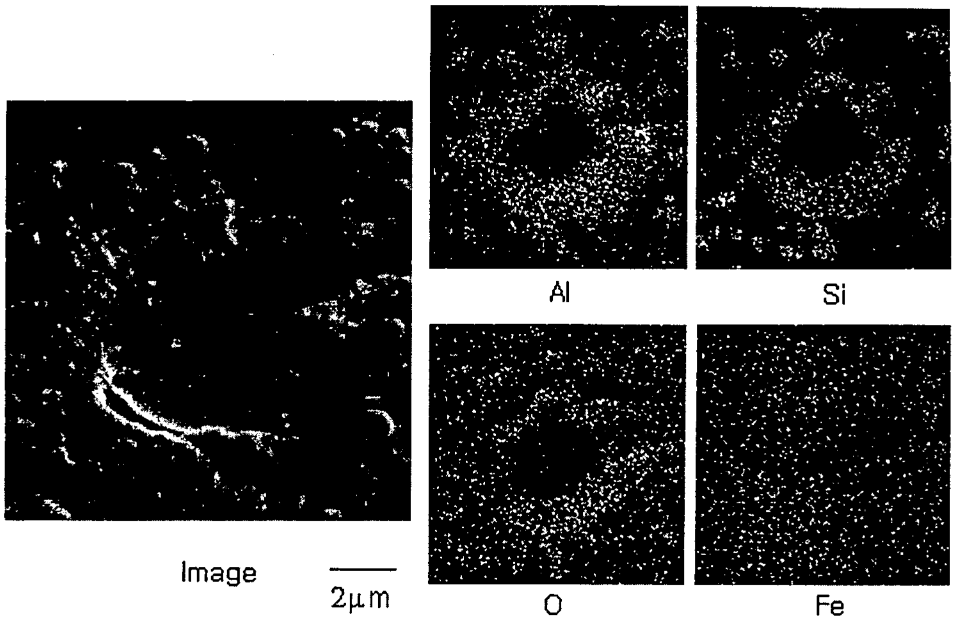

Fig. 5. Compositional mapping of a fly ash particle by an energy dispersive spectrometry.

major components were in the surface area, uneven concentration distribution would not affect the sintering mechanism.

4.2. Numerical analysis

Fast heating in microwave heated system is one of the major advantages. In order to investigate the heating effects of the susceptor and the substituted body, a numerical analysis on the microwave heated system was made. Though direct comparisons are not available, it could give qualitative explanations about the heating mechanism.

Figure 6 shows calculated temperature distributions in the microwave heated system. As seen, one can figure out how heat sources were applied on the sample. It is well understood from the Fig. 6 that in microwave heating system, heat was generated at the location of SiC susceptor inside the cavity and the susceptor showed very concentrated temperature profiles blocked by the Isolite insulating brick. Heat generated at the susceptor propagates from inside of the susceptor to outward. This figure is the case in 8 minutes after putting input power of 750 watts. As seen, white color at the lower left corner represents 714℃ and it went up to 1100℃ within 30 minutes, which was not shown here.

From the results, so far, made in this work, one can conclude in the calculation that microwave heated system could work very well in heating system.

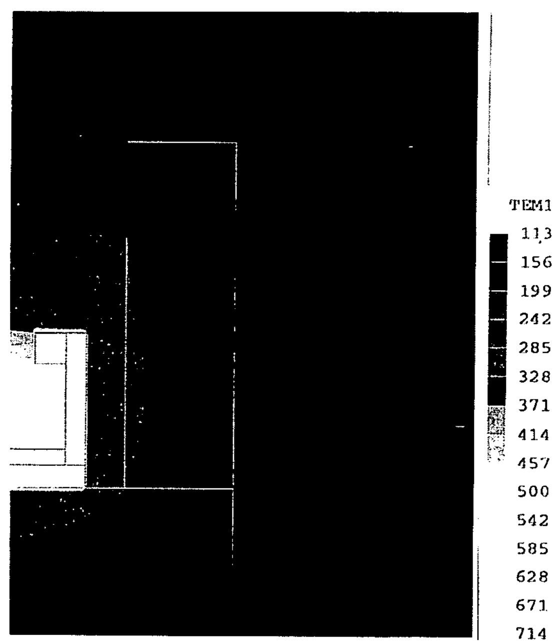

Fig. 6. Calculated temperature distributions in the microwave heated system.

5. Conclusions

1) Sintering of fly ash mixed with clay as 70:30

weight percent ratio was done successfully by using microwave energy.

2) Necking had progressed as sintering time increased from 10 minutes to 50 minutes at 1,150℃ and pore sizes became smaller.

3) Compressive strengths and apparent densities did not change much among the sintered samples at each time intervals

4) Numerical analysis of heat transfer can be an effective method to verify the heat distribution in the cavity during the microwave sintering process.

Acknowledgement

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