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NOTE

# Preparation of $Al_2O_3$ Ceramics by Low Pressure Injection Molding

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Injection molding method is able to prepare green compacts of complicated shape with high precise dimension and good productivity. In this method, there is no need to process mechanically the compacts after molding. It is expected that this method will become a principal technology processing complicated ceramic structural parts. However, the ordinary injection molding method needs such a high pressure as 200 to 2000 Kgf/cm$^2$ for shaping the compacts that the molding apparatus and die must have high wear resistivity against flow of the slurry used. Therefore, the ceramic parts, when the ordinary injection molding is applied for fabrication of many species-small rot parts, become prohibitively expensive. On the contrary, low pressure injection molding gives life of mold tenth or fifteenth times longer than that of ordinary injecrion molding because of applying lower pressure (3 to 5 Kg/cm$^2$). But studies on the fabrication of ceramics by low pressure injection molding using fine powder are scarcely reported[1].

In the present investigation, the possibility of applying low pressure injection molding for processing the high strength-ceramics sintered from raw material of fine particles was examined. Especially the effect of mixing method of the slurry

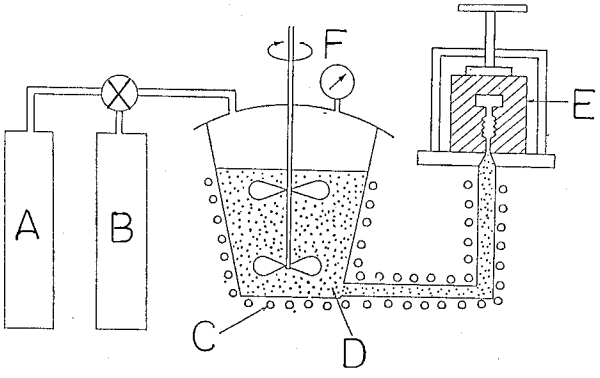


Fig. 1. The schematic diagram of the low pressure injection molding machine
(A) compressor (B) vacuum pump (C) heater (D) slurry
(E) metal mold (F) pressure gauge

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on the flexual strength and its Weibull constant of sintered body was studied.

The green compact was prepared by a low pressure injection molding machine made by Peltzman Co., Ltd. (type MIGL-28) as illustrated in Fig. 1. The mixture of starting powder and binder provides the slurry in the heated tank. The tank and guiding pipe are kept at temperatures in the range from 80°C to 100°C. After the bubbles remained in the slurry are evacuated by the vacuum pump, the slurry is transferred into the metal mold by gas pressure up to 5 $Kgf/cm^2$ of the compressor. The slurry gives rise to a green compact shaped in the water cooled mold.

The $Al_2O_3$ fine powder, AL-160SG, of mean particle diameter of 0.4 $\mu$m, supplied by Shyowa Keikinzoku Co., Ltd. was used after heated at 900°C to remove water adsorbed on the surface of the particles. The organic binder, E-146*, supplied by Chukyo Yushi Co., Ltd. was used.

Fine particles of raw material powder are necessary to be dispersed homogeneously into the binder in order to obtain high strength ceramics. When the size of particles of raw material is smaller than about 1 $\mu$m, the particles tend to make a large agglomerate. In order to disperse uniformly the particles into the organic binder, four different mixing methods were tested; (1) stirrer equipped in the low pressure injection molding apparatus, (2) automatic agate mortar, (3) ceramic three roll mill and (4) ball mill. The content of $Al_2O_3$ powder to the slurry was 85 wt%.

(1) Stirrer; the organic binder of E-146 of 150 g was put in the tank of the hot molding machine and heated at 90°C. After then $Al_2O_3$ powder of 850 g was gradually added into the molten wax and mixed by the stirrer of the hot molding machine for 6 hrs.

(2) Automatic agate mortar; Ishikawa-type automatic agate mortar was used. While the binder was melted by a hot blower, $Al_2O_3$ powder was added and mixed for 4 hrs.

(3) Ceramic three roll mill; the ceramic three roll mill, type 9×18 C (the ratio of rotation rate is 1:3:9) produced by Inoue Seisakusho Co., Ltd. was used. Small amount of methyl ethyl keton was added to the slurry premixed by the automatic mortar to give the slurry adequate plasticity. The slurry, thus prepared was passed through the rolls four times.

(4) Ball mill; $Al_2O_3$ ball mill Type HD pot-mill B (capacity 7300 ml) produced by Nippon Kagaku Togyo Co., Ltd. was used. $Al_2O_3$ powder of 1700 g, the binder of 300 g and methyl ethyl keton of 1000 ml were introduced in the pot with $Al_2O_3$ balls, and milled for 24 hrs. Methyl ethyl keton makes a role of solving the wax. After milling, the solvent was evaporated by heating the suspension at 70°C.

Fibers of diameter of about 100 $\mu$m were prepared from the molten slurry kept at 80°C, by pulling abruptly a glass stick from the slurry after being contacted with it. The presence of agglomerate on the surface of the fiber was detected by naked eye or optical microscope.

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* E-146 involves 70wt% of normal paraffin (molecular weight 300 to 500), 20wt% of branched paraffin (molecular weight 500 to 700) and 10wt% of dispersing agent. The m.p. of this wax is 52°C.

The results were summarized in Table 1.

A lot of agglomerates were observed in the fiber of the slurry prepared by the stirrer mixing method and it was impossible to make a long fiber from the slurry. On the other hand, no agglomerate was detected over the surface of the fiber pulled

Table 1 Appearance of four kinds of slurries

| Mixing method | Mixing time | Fiber formation | Agglomerates |
|---|---|---|---|
| Stirrer | 6 hr | Difficult | Observed abundantly |
| Agate mortar | 4 hr | Easy | Trace |
| Ceramic three roll mill | 15 min | Easy | Trace |
| Ball mill | 24 hr | Easy | Not detected |

from the slurry prepared by the ball mill mixing method. The slurries prepared from the automatic agate mortar and the ceramic three roll mill showed a trace of agglomerate on the surface of the fiber. It is concluded that the ball mill mixing method prepares the most uniform slurry of the four slurries above described.

The viscosities of their slurries were measured with temperature from 65°C to 90°C by B Type Viscometer produced by Tokyo Keisoku Co., Ltd.

Fig. 2 shows the measured viscosity of the slurries. The slurry prepared by

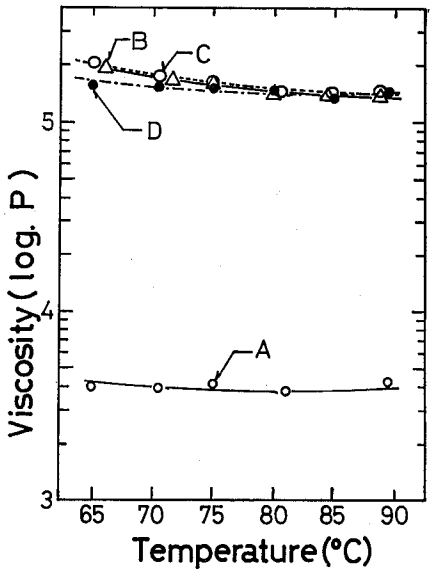


Fig. 2. Viscosity of the four kinds of slurries
(A) stirrer (B) automatic agate mortar
(C) ceramic three roll mill (D) ball mill

the stirrer mixing method showed the lowest viscosity while the other slurries showed near the same high value. The more uniformly dispersed the particles are, the higher the viscosity of the slurry is. Therefore, the slurries prepared by above methods except the stirrer mixing one were thought to be homogeneous so far observed

from viscosity.

The green compacts were molded by using two kinds of mold; one was a plate of 50 mm×50 mm×3 mmt, and the other was one of 50 mm×50 mm×5 mmt. The conditions of molding are shown in Table 2.

Table 2 Conditions of molding

| Thickness of plate | Temperature of tank | Temperature of pipe | Molding time | Molding pressure |
|---|---|---|---|---|
| 3 mm | 100°C | 90°C | 20 sec | 5 Kgf/cm² |
| 5 mm | 100°C | 90°C | 30 sec | 5 Kgf/cm² |

Table 3 Specific weight of green compact

| Mixing method | Specific weight (gr/cm²) | |
|---|---|---|
| | Thickness of mold | |
| | 3 mm | 5 mm |
| Stirrer | 2.282 | 2.284 |
| Automatic agate mortar | 2.592 | 2.567 |
| Ceramic three roll mill | 2.662 | 2.661 |
| Ball mill | 2.614 | 2.603 |

The specific weight of the green compact was measured the by the Archimedes method.

The result was shown in Table 3. The specific weight of stirrer-mixed green compact was the smallest and those of the others were near the same.

The curves of thermo gravimetric analysis (TGA) of the green compacts were measured. The heat program of dewaxing process was determined so that the heating rate might be small in the range where a large decrease of TGA was observed. The green compacts were dewaxed in the $Al_2O_3$ powder and sintered, according to the following heat program. The temperature program for dewaxing was as follows; RT-200°C: heating rate 5°C/h, 200°C: holding time 0.1 h, 200°C–400°C: heating rate 2°C/h and 400°C–900°C: heating rate 5°C/h. The temperature program for sintering was as follows; RT-1200°C: heating time 3 h, 1200°C–1700°C: heating time 1.5 h, 1700°C: holding time 3 h, and 1700°C–1200°C: cooling time 1 h. Neither crack nor other defects were observed in the dewaxed specimens and also in the sintered specimens by naked eye.

The flexual surface of the test piece was finished by diamond paste of granular

Table 4 Flexual strength of sintered $Al_2O_3$ ceramics and their Weibull constants

| Mixing method | Sintering temperature °C | Average of flexual strength Kgf/mm² | Number of specimen | Weibull constant |
|---|---|---|---|---|
| Stirrer | 1700 | 22.1 | 24 | 3.7 |
| Automatic agate mortar | 1700 | 28.3 | 26 | 4.6 |
| Ceramic three roll mill | 1700 | 32.5 | 47 | 5.2 |
| Ball mill | 1700 | 40.0 | 26 | 6.9 |

diameter under 1 $\mu$m, after grinding it by #400 diamond tool. The flexual strength of $Al_2O_3$ ceramics sintered at 1700°C for 3 hrs was measured by three point bending test at room temperature and Weibull constants were calculated.

The results were shown in Table 4.

The flexual strength of sintered specimen from the ball mill mixing method shows the highest value 40 Kgf/mm$^2$, while that of the test pieces from the stirrer mixing method shows the lowest value 22.1 Kgf/mm$^2$. Similarly, the sintered specimen from the ball mill mixing method shows the highest Weibull constant 6.9 while that from the stirrer mixing method shows the lowest value 3.7. The high Weibull constant results from small probability of presence of defects which give rise to fracture under an external stress. The sintered specimen from the ceramic three roll mill shows the second highest value of the flexual strength and Weibull constant. The order of homogeneity of the slurry as shown in Table 1 is in good coincidence with that of Weibull constant.

From these experimental results, it is concluded that the homogeneously dispersed slurry prepared by the ball mill mixing method gives the high strength sintered $Al_2O_3$ ceramics with high Weibull constant.

## REFERENCE

(1) K. Miyamoto, H. Miyamoto, Y. Takahashi and H. Taguchi, Annual meeting of the Japan Society of Powder and Powder Metallurgy, May, 27–29, 1986, p. 164.