Wet Vibration Pressing of Alumina

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Abstract

The wet vibration pressing with alumina soft-mud or slurry has been investigated in porous molds. The applied pressure could be reduced to under one-tenth of that in conventional metal die pressing with dry alumina powders, because the moisture content is high enough to allow freedom of flow of alumina powders. About 60 seconds of vibration was necessary to achieve the maximum green density value. The forming factors of both amplitude and frequency of vibration had little influence on the green density of compacts when it was over that period.

In this process with alumina soft-mud material, the green density of compacts reached 60 percent, near to that of conventional die pressing in relative density. The green density increased remarkably in the process with alumina slurry material, and it reached 66 percent or more.

1. Introduction

The forming of alumina powder is accomplished by many methodes such as dry pressing, hydrostatic pressing, slip casting and vibration casting. The use of vibration to assist the packing of dry ceramic powders is well known.

A number of studies on the vibration compacting of dry ceramic powders under low load have been published.[1~4] W.C.Bell et al., using a pneumatic vibrator with low frequency vibration, reported that alumina specimens had properties which were approximately the same as specimens formed by dry pressing followed by hydrostatic re-pressing.[1] I.G.Shatalova et al., using a mechanical vibrator with a constant frequency and amplitude, compacted TiC, WC powders to acceptable density with pressure of 100 times less than conventional compacting pressure.[2]

Y.Ozaki et al. also investigated the compacting of alumina powder using a mechanical vibrator.[3] J.L.Brackpool et al. reported that the vibratory compacting of copper powder produced compacts of improved uniformity and green density.[4] Furthermore, D.McFetrige et al. studied vibration pressing of dry ceramic powders under high load, and concluded that the achieved density increased as G force increased and for each G force there is an optimum frequency range.[5]

But, these vibration compacting and pressing methods of dry ceramic powders have been not widely used for industrial purposes, because the green density of the compacts of fine powders formed by these methods is near to that of conventional die pressing, and it is greatly influenced by many forming factors such as amplitude, frequency of vibration, vibration time and applied pressure.

Thus, the wet vibration pressing under comparative low load with alumina soft-mud or slurry material has been investigated. This method is a new plastic pressing with vibration, or it is a process which combined vibration casting and die pressing.

2. Experimental

2.1 Alumina materials

Sumitomo Chemical Industry Co.,Ltd. AES-12 alumina fine powder of 0.4 μm in mean particle size was used in this investigation. The chemical composition and properties of alumina powder were shown in Table 1.

Alumina soft-mud was firstly prepared by manually kneading of both alumina powder and 20 wt.% of binder in a mortar. The binder consisted of a solution of nine parts 2 wt.% P.V.A. sol. and a part of surface-active agent by weight.

Secondly, alumina slurry material was prepared by blending of alumina powder and 16 wt.% of binder in a ball mill with zirconia ball for 24 h. The binder was a solution of 2 % P.V.A. sol. with a small quantity of dispersant, plasticizer, lubricant, surface-active agent and antifoaming of Dai-ichi Kyogo Seiyaku Co., Ltd. products.

2.2 Experimental apparatus and procedure

The vibration pressing machine is shown schematically in Fig.1. It consists of vibration top plate with an oil cylinder and bottom plate, both of which are fixed by four poles, and four top and bottom springs supported two plates. The mechanical vibrator (Uras Vibrator KED-3-2, Vibrating force 300 kg, Murakami Seiki, Mfg. Co., Ltd.) is attached to the bottom of a vibration plate and it consists essentially of two contra-rotating shafts with out-of-balance weights at each end. The die set in vibration plates has a cavity 20 mm in diameter and 70 mm in depth and is made from porous molds. Top and bottom punches are also made from porous molds which are able to adsorb enough binder for forming of compacts. The porous molds was composed of plaster or graphite electrode.

Preliminary experiments were carried out to establish operating techniques and to obtain optimum quantity of binder and optimum forming conditions. The vibration and pressure were applied simultaneously. It takes 60 sec to reach a fixed pressure after die was set in vibration plates. The standard forming vibration time after a fixed prssure was 60 sec, amplitude was 0.9 mm, frequency of vibration was 60 Hz, and applied pressure was 10 MPa. The amplitude was determined by the aid of a pocketable vibration meter (Riovibro VM-63, Rion Co., Ltd.) and the frequency of vibration was done by a digital hand tachometer (HT-441, Ono Sokki Co., Ltd.). Obtained specimens were 20 mm in diameter and about 20 mm in length.

3. Results and discussion

3.1 Wet vibration pressing of alumina soft-mud material

Firstly, the effect of applied pressure on the green density of formed specimens was investigated using alumina soft-mud material at a constant forming condition on which amplitude was 0.9 mm, frequency was 60 Hz and vibration time was 60 sec. The relation obtained between green density of specimens and applied pressure was shown in Fig. 2. It also showed the variation of bulk density of specimens sintered at 1500 ℃ for 5 h with various applied pressures. Conventioal metal die pressing was carried out to compare this study using dry alumina powder with 5 wt.% of 2 % P.V.A. solution.

Both green density and sintered density of specimens were slightly influenced by applied pressure, although conventional die pressing was greatly influenced by that. The green density of compacts in this study, using low pressure of one-tenth to one-twentieth of conventional die pressing, was equivalent or more to that of die pressing which was about 60 percent in relative density. The sintered density of specimens was also near to that of die pressing. Therefore, compacting pressure could be greatly reduced by applying vibration in this study

It is supposed from these results that alumina powders easily moved by thixotropic flow owing to vibration and that they packed to the remarkable high density because the moisture content is high enough to allow freedom of alumina powders. Therefore, bridge phenomena caused by friction resistance among powders and between powders and die wall could be greatly reduced.

The relation between the green density and sintered density of specimens and vibration time after fixed applied pressure was shown in Fig. 3. The forming was carried out at a constant condition on which applied pressure was 10 MPa, frequency was 60 Hz and amplitude was 0.9 mm. The green density increased with the increase of vibration time, though it was not remarkably affected by the time. Then, about 60 sec on vibration was necessary to achieve the fixed green density value in this investigation, that is, it takes 60 sec for porous molds to absorb enough moisture in binder used for forming of compacts.

Fig. 4 showed the effect of frequency of vibration on both green density and sintered density of specimens at a constant condition under 10 MPa of applied pressure, 60 sec of vibration time and 0.9 mm of amplitude. The frequency of vibration had little effect on density of specimens, although dry vibration pressing had an optimum frequency range for each ceramic powder. Furthermore, the amplitude also had little effect on density of specimens at more than 0.3 mm.

Fig. 5 showed the Vickers hardness of surface of sintered specimens at various sintering temperatures for 5 h. The surface hardness increased with the increase of firing temperature, and it was higher than that of die pressing at 100 MPa, although green density of compacts was near to that die pressing.

3.2 Wet vibration pressing of alumina slurry material

Although wet vibration pressing of alumina soft-mud material could greatly reduce the compacting pressure, it could not greatly increase the green density of specimens, compared with conventional metal die pressing. Then, by using alumina slurry the study has been carried out in the same methods. This method is a process which combined vibration casting and die pressing.

The fluidity of prepared alumina slurry material was investigated before vibration pressing. The fluidity of 0.5 g of prepared slurry was examined by measuring of flowed area on slide glass before and after vibration. Fig. 6 showed the relation between ratio of flowed area of slurry and vibration time at a constant condition under 0.9 mm of amplitude, 60 Hz of frequency of vibration and no load. The flowed area of slurry increased with the increase of vibration time, and it was trebled at 30 sec and became saturated after that time. Aspects of flowed slurry before and after vibration for 30 sec were shown in Fig. 7. The flowed area also increased with the increase of amplitude. On the other hand, soft-mud alumina material was not flowed by use of vibration. Therefore, it is thought that alumina powders in this study could be packed to higher density in comparison with that of previous soft-mud material owing to more thixotropic flow by use of vibration.

The relation between the green density of specimens and applied pressure was shown in Fig. 8. The forming was carried out at a constant condition under 60 sec of vibration time, 60 Hz of frequency of vibration and 0.9 mm of amplitude. The applied pressure had little effect on the green density of specimens. The green density increased remarkably in this process with alumina slurry material, and it reached 66 % or more in relative density.

On other forming factors, about 60 sec of vibration time, equal to that of previous investigation with soft-mud material, was necessary to achieve the maximum green density value. Furthermore, the frequency of vibration had little effect on green density of specimens at more than 30 Hz, and the amplitude also had little effect on density at more than 0.3 mm. These little effects on the green density of specimens were shown in Fig. 9 and Fig. 10.

The bulk density of specimens with slurry material sintered at 1500 ℃ for 5 h naturally increased in comparison with that of specimens with soft-mud material. Fig.11 showed the variation of apparent porosity of specimens at 1500℃ with various applied pressures. The apparent porosity greatly decreased in comparison with that of previous investigation with soft-mud material. And the firing shrinkage after sintering was about 10 %, although that of conventional die pressing was about 14.5 %.

But, slight crack and hollow were seen on the top surface of fired specimens with about 1 mm of depth, though those defects disappeared by cutting of top surface. These defects gave a few variations on the apparent porosity of fired specimens in Fig. 11. As these defects were removed, the apparent porosity of specimens approached to 0 %.

It is thought that these defects depend on insufficient packing of alumina powders, because those powders used for this investigation were material for die pressing and was not thixotropic powders. Therefore, both alumina powders and the binder were reexamined in order to prepare more thixotropic slurry. Those detailed results are scheduled to report in next paper. When the binder was changed from P.V.A. solution to emulsion type binder, the follow on top surface of sintered specimens reduced and the crack disappeared, although the green density of specimens was not changed. When both alumina thixotropic powders for slip casting and emulsion type binder were used, the green density of specimens reached 70 % or more in relative density and the defects on top surface of fired specimens were disappeared.

4. Summary

The wet vibration pressing with alumina soft-mud or slurry material was investigated in porous molds.

(1) The applied pressure could be reduced to under one-tenth of conventional die pressing.

(2) The forming factors of applied pressure, time of vibration, amplitude and frequency of vibration had little effect on the green density of specimens at more than 10 MPa, 60 sec, 0.3 mm and 30 Hz respectively.

(3) In this vibration pressing with soft-mud material, the green density of compacts was was about 60 %, near to that of die pressing in relative density.

(4) In case of slurry material, the green density increased remarkably and it reached 66 % or more, but sintred specimens had defects of slight crack and hollow on their top surface.

(5) This process with low load may be valid for forming of large, thick and complicated shape products for structural ceramics.

References

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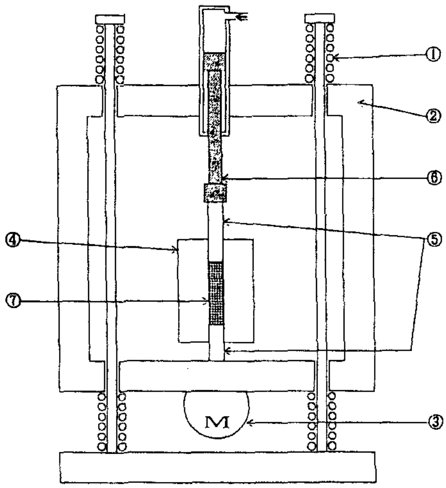

① Spring ② Vibration plate
③ Vibrator ④ Die
⑤ Punch ⑥ Oil cylinder
⑦ Alumina material

Fig. 1 Schematic layout of vibration pressing machine.

Table 1. Characterization of alumina powder.

Crystal tipe	α
True density (g/cm³)	3.95
Chemical composition (%)	
Al_2O_3	99.9
Fe_2O_3	0.01
SiO_2	0.06
Na_2O	0.05
Particle size (μm)	
20%	0.3
50%	0.4
90%	0.8
mean	0.4

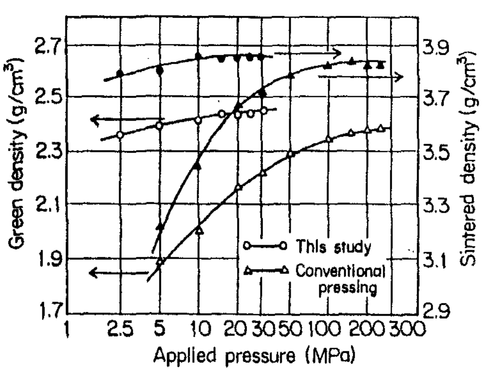

Fig. 2. Relation between bulk density of specimens and applied pressure.

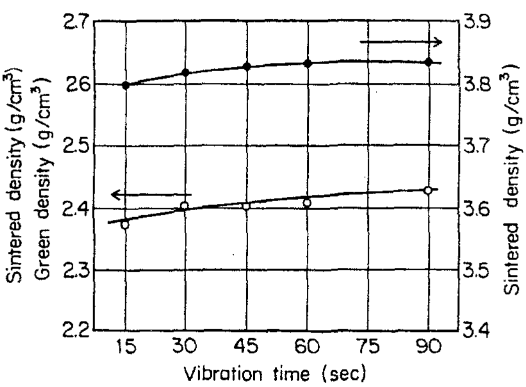

Fig. 3. Relation between bulk density of specimens and vibration time.

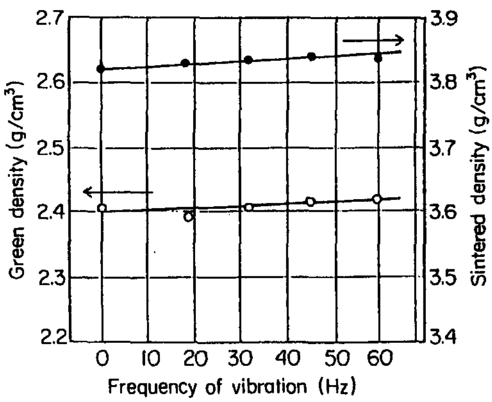

Fig. 4. Effect of frequency of vibration on the green density and sintered density of specimens.

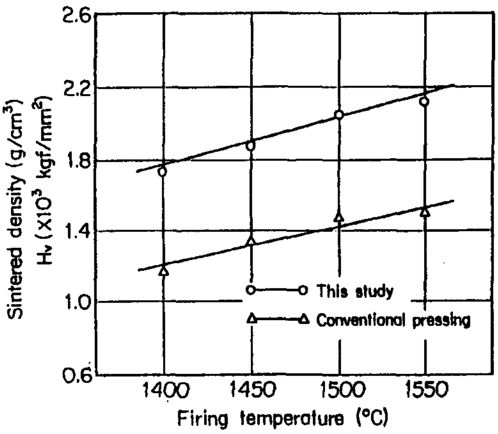

Fig. 5. Vickers hardness of surface of sintered specimens.

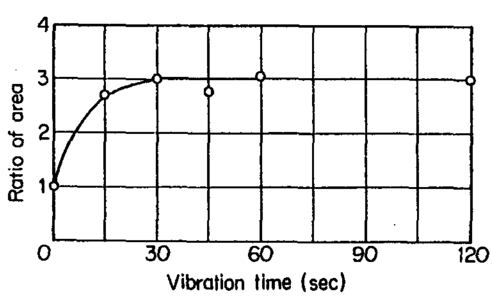

Fig. 6. Relation between ratio of flowed area of slurry and vibration time.

0 sec 30 sec

Fig. 7. Aspect of flowed slurry before and after vibration.

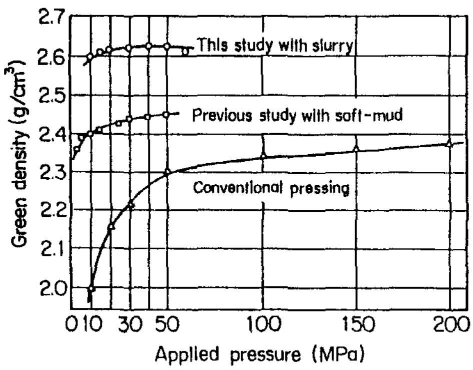

Fig. 8. Relation between green density of specimens and applied pressure.

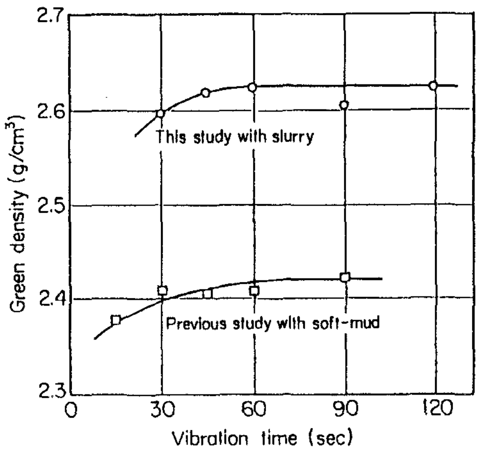

Fig. 9. Effect of vibration time on the green density of alumina compacts.

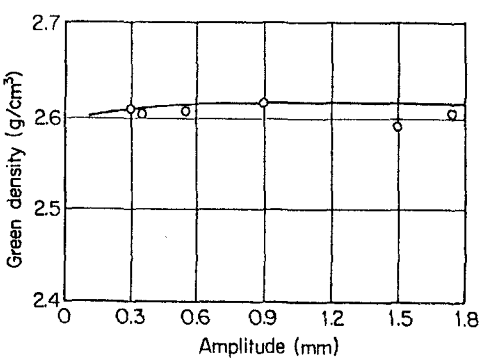

Fig. 10. Effect of amplitude on the green density of alumina compacts.

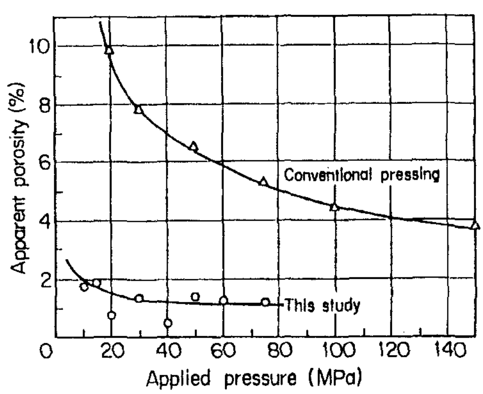

Fig. 11. Variation of apparent porosity of sintered specimens with various applied pressures.