Development of Injection Molding Technology using Aluminum sludge

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Abstract
Aluminum sludge is a precipitation yielded during the acid/ alkari neutralizing process of waste fluid derived from the surface treatment of aluminum sash. By heating the sludge at 1573 K for 2 hours, the sludge is transfered into α alumina structure. Therefore, we attempted to apply the alumina sludge as the ceramic material for injection molding. Then we investigated the optimum conditions in kneading, molding, degreasing and firing processes to get a consistently high strength of the product by using design of experiments. We could ascertain the specific factors of firing temperature and cylinder temperature which particularly affected the bending strength of the product. As a result, we were able to attain sufficient strength in the product using sludge by injection molding process.
Keywords:*sludge, aluminum, alumina, ceramics, injection molding*

1. Introduction

Injection molding has an advantage in the making of a complicated 3-D product. Also ceramics material is superior to metal in many ways including hardness, resistance to heat and mechanical strength. Because of these advantages, the product manufactured by the ceramics injection molding process is mainly used as a heat engine component in automobiles. In general, the commercial industrial ceramics material is extremely expensive because it requires a high purity in its chemical components and micro meter size of particle. Its high price is the main obstacle to the wide use of fine ceramics for popular industrial products.

From the point of view that clay is composed of particles which also have a micro meter size in diameter, we should try their application in the injection molding process. By using a excellent fine powder derived from chemical PH treatment of clay mixed with water and several optimum binders, the sintered products have enough hardness and high bending strength.[1)]

Incidentally, the disgarding of sludge is generally performed at areas prescribed by public institutions. Various issues such as the selection of dumping sites and the high cost involved are becoming large problems.[2)] Therefore, an effective technique for using sludge as an industrial material is expected to be developed. Because sludge contains mainly alumina components, there is a possibility of transforming the sludge into a hopeful ceramics material by performing heat treatment at high temperatures. Accordingly, we are investigating the application of injection molding to the sludge.

The ceramic injection molding process consists mainly of four stages: the kneading of the mixture, the molding process, the degreasing process and the burning process. Because each stage has many parameters, we must perform a large number of experiments changing many conditions in the process. Therefore, in order to efficiently obtain a high strength product, the design of the experiment was chosen to determine the optimum condition.

Finally, by setting the optimum condition, we were able to ascertain the effect of each factor and level at each stage, to the bending strength and to produce a high quality product using sludge.

2. SN ratio analysis

The optimization of bending strength in the product was determined by comparing the SN ratio based on the design of experiments. The S(Signal)N(Noise) ratios were calculated by using the following equation:[3)]

$$\eta = -10 \times \log\left[\frac{1}{n}\left(\frac{1}{y_1^2} + \frac{1}{y_2^2} + \cdots + \frac{1}{y_n^2}\right)\right] \qquad (1)$$

η :SN ratio (dB) Yn:Measuring value n:Number of data

A larger value indicates a better condition for the strength of the product, therefore, this factor indicates a strong effect to the strength.

A smaller value indicates a worse condition for the product, because the noise to signal ratio becomes large, and that causes a reduction in the strength of the product. Accordingly, the optimum condition by using this method could be found .

3. Experimental material and method

3.1 Aluminum sludge

First, aluminum sludge was cleaned sufficiently by stirring the water mixed with sludge. After solar drying the sludge, the water in the sludge was completely removed by firing at 423 K in the furnace. Then the sludge was milled for several hours. After the sludge was fired at 1573 K for 2 hours, it was them cooled gradually in the furnace.

3.2Injection molding

The ceramic injection molding process mainly consists of kneading, molding, degreasing and firing. In the molding process, it is necessary to choose the binders because ceramic powders cannot flow. Table 1 shows the ingredient ratio of binders for making pellets. The mixing of the sludge and binders was performed with a twin screw type kneader at 403 K for 2 hours. Figure 1 shows the green product molded by the injection machine (Nissei Jushi Kogyou Company). The test piece was prepared by cutting areas A and B as shown in Fig.1. Then, degreasing using an SHKS-1 (Fine company) and Firing using an SB-1415C (Motoyama company) were performed.

3.3Estimation of product

The three point bending test was carried out by using an instoron type test machine with a cross head speed of 0.5mm/min. Bending strength(σ) was calculated as follows:

$$\sigma = 8PL/\pi d^3$$

P: Breaking load(N) d: Diameter of test piece(mm)
L: Distance between two supporting points(mm)

For the purpose of conveniently knowing the pore ratio in the product, the water absorption ratio was measured by calculating the difference of weight before and after been soaked in boiling water for 3 hours as follows:

$$\text{Water absorption}(\%) = (W_2 - W_1)/W_1$$

W_1:Weight of the product dried at 393 K in the furnace
W_2:Weight of the product soaked in boiling water for 3 hours

Table 1 Ingredient ratio of compound for injection molding of sludge. DBP and EVA are Dibutyl Phthalate and Ethylene Vinylacetate Copolymer, respectively

Material	(mass%)
Sludge	72.0
DBP	6.6
EVA	7.9
Paraffin wax	7.0
Zinc stearin	6.5

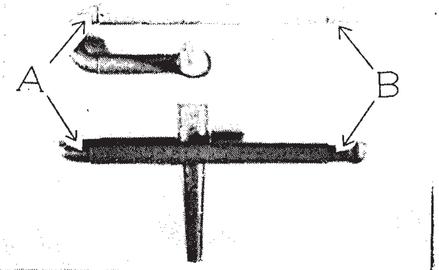

Fig. 1 Green product.

4. Experimental result and discussion

4.1 Aluminum Sludge

In its original state the sludge includes 77 % water , consists of beimite components, and transforms into various structures through heat treatment. Therefore, the sludge was fired at 1573 K for 2 hours. The diffraction profile of X ray analysis for the sludge is shown in Figure.2. α alumina corundum was used as a material for comparison. The upper diagram shows the X-ray diffraction pattern for the sludge. The upper portion of the lower diagram shows the peak points for the sludge, while the lower portion shows the peak points for the corundum. Because the peak points for both materials are in agreement, it can be ascertained that the sludge structure is mostly α alumina. Aluminum sludge will hereafter be referred to as α sludge.

Measuring the grain size distribution (as shown in Fig.3) of the α sludge powder after being crushed by a mill revealed that it is composed of a variety of sizes of particles with the average diameter being 6.8 μm.

4.2Bending strength of injection molding product

Because the ceramic injection molding process consists of kneading, molding, degreasing and firing, it is difficult to find the optimum condition for obtaining a consistently high bending strength. Therefore, we adopted the design of experiments to determine the optimum condition effectively. To use this analysis, at this juncture, the most important point is to choose the factors and the corresponding level settings. The seven factors and two or three levels in each factor were chosen as shown in Table 2. Burning temperature, rising time required to reach the setting temperature for the burning process, injection pressure, molding temperature, cylinder temperature, holding time for high pressure for molding process, and degreasing speed for the degreasing process were chosen as control factors.

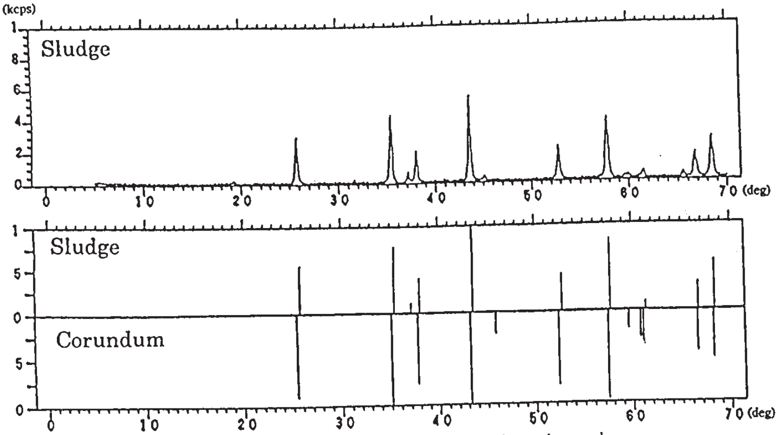

Fig. 2 Diffraction profile of X-ray analysis for sludge and corundum.

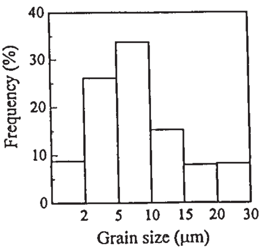

Fig. 3 Grain size distribution of aluminum sludge.

Table 2 Controlling factors and those level for injection molding and firing

	Factors	Level		
		1	2	3
C	Injection pressure (MPa)	5.8	8.8	11.8
D	Mold temperature (K)	307	313	319
E	Cylinder temperature (K)	373	393	413
F	Holding time (s)	5	15	25
G	Degreasing speed (K/h)	5	30	55
B	Rising temp. time (h)	2	4	6
A	Firing temperature (K)	1773	1873	
e	Error			

Table 3 Orthogonal array L_{18} in bending test

	A	B	C	D	E	e	F	G
1	1	1	1	1	1	1	1	1
2	1	1	2	2	2	2	2	2
3	1	1	3	3	3	3	3	3
4	1	2	1	1	2	2	3	3
5	1	2	2	2	3	3	1	1
6	1	2	3	3	1	1	2	2
7	1	3	1	2	1	3	2	3
8	1	3	2	3	2	1	3	1
9	1	3	3	1	3	2	1	2
10	2	1	1	3	3	2	2	1
11	2	1	2	1	1	3	3	2
12	2	1	3	2	2	1	1	3
13	2	2	1	2	3	1	3	2
14	2	2	2	3	1	2	1	3
15	2	2	3	1	2	3	2	1
16	2	3	1	3	2	3	1	2
17	2	3	2	1	3	1	2	3
18	2	3	3	2	1	2	3	1

Table 4 Experimental result on bending strength of fired products

(MPa)

	1	2	3	4	5	6
1	57.7	57.9	64.8	67.2	76.6	64.2
2	42.3	58.6	69.2	78.1	35.9	81.9
3	89.2	0	0	64.3	62.5	93.7
4	72.5	76.1	89.1	84.7	69.2	73.0
5	25.8	74.8	72.4	22.2	71.4	49.2
6	25.7	0	0	67.5	63.8	63.3
7	0	48.5	0	90.0	25.8	83.4
8	84.0	89.0	85.0	90.1	98.5	82.7
9	74.1	59.8	75.9	72.9	76.7	92.2
10	167.4	234.4	231.6	257.7	225.8	291.4
11	253.6	149.2	294.5	149.2	163.5	0
12	184.8	244.9	197.8	273.9	207.6	202.1
13	0	202.3	267.0	225.6	65.5	219.8
14	233.6	0	0	0	228.7	0
15	217.5	249.0	197.6	251.6	193.1	219.1
16	243.8	268.4	274.5	228.9	240.9	261.4
17	200.0	215.3	246.0	208.8	244.6	0
18	185.3	189.6	258.1	243.7	204.9	217.1

Table 5 SN ratio transformed from experimental data

(dB)

	1	2	3	4	5	6
1	35.22	35.25	36.23	36.55	37.68	36.14
2	32.53	35.35	36.80	37.85	31.11	38.26
3	39.00	25.19	25.19	36.16	35.92	39.44
4	37.21	37.62	38.99	38.55	36.80	37.26
5	28.22	37.47	37.19	26.94	37.08	33.84
6	28.19	25.19	25.19	36.58	36.10	36.03
7	25.19	33.71	25.19	39.08	28.22	38.43
8	38.49	38.98	38.59	39.09	39.87	38.36
9	37.39	35.53	37.61	37.25	37.69	39.29
10	44.47	47.40	47.29	48.22	47.07	49.29
11	48.08	43.48	49.38	43.48	44.27	25.19
12	45.34	47.78	45.92	48.75	46.35	46.11
13	25.19	46.12	48.53	47.07	36.33	46.84
14	47.37	25.19	25.19	25.19	47.19	25.19
15	46.75	47.92	45.91	48.02	45.72	46.81
16	47.74	48.57	48.77	47.19	47.64	48.35
17	46.02	46.66	47.82	46.39	47.77	25.19
18	45.36	45.56	48.24	47.74	46.23	46.73

Table 6 Dispertion analysis. f, S, V, F_0 and ρ are freedom, squared sum, variance, F value and contribution ratio, respectively

	f	S	V	F_0	ρ(%)
C Injection pressure	2	30.70	15.35	3.71	4
D Mold temperature	2	13.31	6.66	1.61	1
E Cylinder temperature	2	90.88	45.44	11.00*	14
F Holding time	2	3.78	1.89	0.46	0
G Degreasing speed	2	47.77	23.89	5.78	7
B Rising temp. time	2	46.91	23.45	5.68	6
A Firing temperature	1	355.62	355.62	86.06*	58
e error	4	16.53	4.13		11
Total	17	605.50	—		100

* Significant

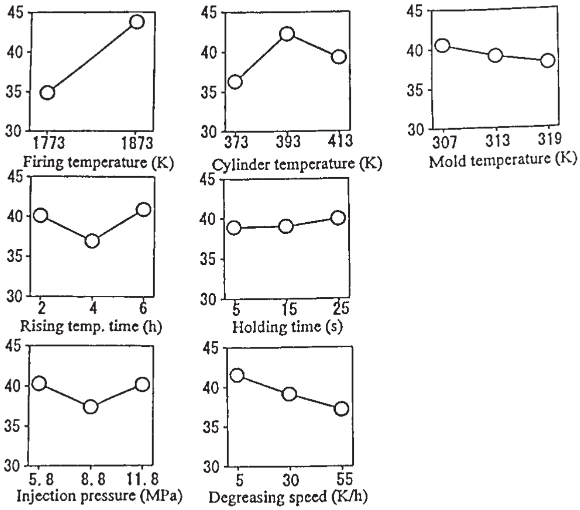

Fig. 4 SN ratio on each level of factor.

The e symbol in Table 2 indicates error factor. The experiments were performed with eighteen combinations based on L_{18} as shown in Table 3. The three point bending strength result is shown in Table 4. Table 5 shows the SN ratio transformed from the bending strength. The analysis of variance using these data is shown in Table 6. The symbols f, S, V, F and ρ in Table 6 indicate freedom, variance, dispersion, F value and contribution ratio, respectively. From the F test, the factors of burning temperature and cylinder temperature were judged as significant through rejection ratio of 5%. Contribution ratio shows the burning temperature with the highest value of 58% and cylinder temperature with the next highest at 14%. These results suggest that the bending strength of product depends on the burning temperature which affects the degree of particle sintering. And cylinder temperature affects the flow binders melting state when pushed by screw during the molding process. Therefore, SN ratios in each factor were compared as shown in Fig.4. From the comparison between 1773K and 1873K in burning temperature, the SN ratio at 1873K shows an improvement of 9dB, and the high temperature is desired as a firing temperature. In the cylinder temperature, SN ratio at 393K shows the peak point which demonstrates that there exists a optimum cylinder temperature for viscosity. The other factors show little difference among the levels. Next, the bending strength of the product was estimated by using the optimum condition based on the significant factors which are burning temperature and cylinder temperature.

$$\hat{\mu} = A_2 + E_2 - H = 43.72 + 42.26 - 39.28 = 46.7\,(dB)$$

$$\mu = \hat{\mu} \pm \sqrt{f_e^1(0.05) \times \frac{V_e}{n_e}} = 46.7 \pm \sqrt{7.71 \times \frac{4.13}{18/4}}$$

$$= 46.7 \pm 2.83\,(dB)$$

When the experiments using the optimum condition were performed, the bending strength shows 45.81dB (195.3MPa). From this result, the optimum condition confirmed the reliability.

4.3 Effect of burning temperature on quality of the product

As the results from the analysis of variance that burning temperature mainly affects the bending strength became obvious, burning temperature can naturally be considered to affect other qualities of the product. Therefore, the affect of burning temperature was investigated by changing the burning temperature alone, while fixing the conditions of the other factors. Figure 5 shows the morphology of the green product and the products fired at various temperatures. From a comparison of the products, we were able to observe the shrinkage of size as the temperature rose. The average diameters measured on three points of each test piece were compared in order to ascertain the amount of shrinkage of the products. Figure 6 shows the relation between shrinkage and firing temperature. The shrinkage increases linearly as the firing temperature rises. Next the comparison of the water absorption ratio was performed in order to know the pore ratio by measuring the weight before and after soaking the product in boiling water for 3 hours. Figure 7 shows the relation between water absorption and firing temperature. As the burning temperature rises, water absorption decreases linearly. Particularly, because the water absorption between temperatures of 1773K and 1823K changed rapidly from 18% to 5%, the sintering phenomena of the particles at 1823K and above were considered to be achieved rapidly. From the result that the water absorption is 1% at the burning temperature of 1873K, pores on the surface of the product were virtually absent and the density of the product was 3.56 Mg/m^3. Figure 8 shows the relation between the bending strength and firing temperature.

As the firing temperature rises, the bending strength increases drastically at temperatures of 1823K and above, although bending strength increases only gradually up to a temperature of 1773K. The bending strength shows the highest value at 1873K. The high bending strength at 1823K and above is due to the smoothing sintering phenomena. Figure 9 shows the microphotography of the bending fracture surface of the product when changing the firing temperature. As the features of α sludge remained between temperature of 1673K and 1773K, there is no evidence of sintering phenomena. Although we are able to observe a few sintering phenomena at 1823K, some pores remain. Because of the existence a large number of flat planes at 1873K,we could confirm that the smoothing sintering phenomena had occurred. From these results, the improvement of the bending strength of injection molding products using α sludges appeared at the sintering temperature of 1823K, and drastically increased at 1873K.

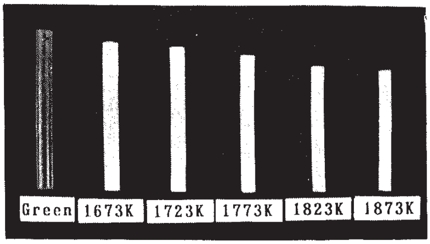

Fig. 5 Morphology of green product and product fired at various temperature.

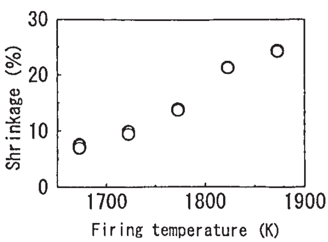

Fig. 6 Relation between shrinkage and firing temperature.

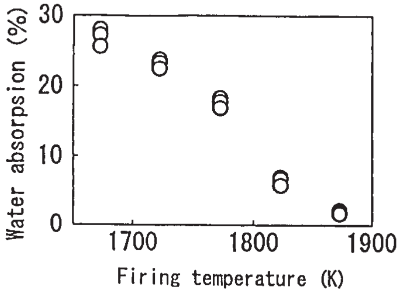

Fig. 7 Relation between water absorption and firing temperature.

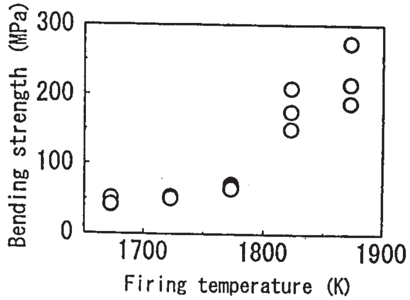

Fig. 8 Relation between bending strength and firing temperature.

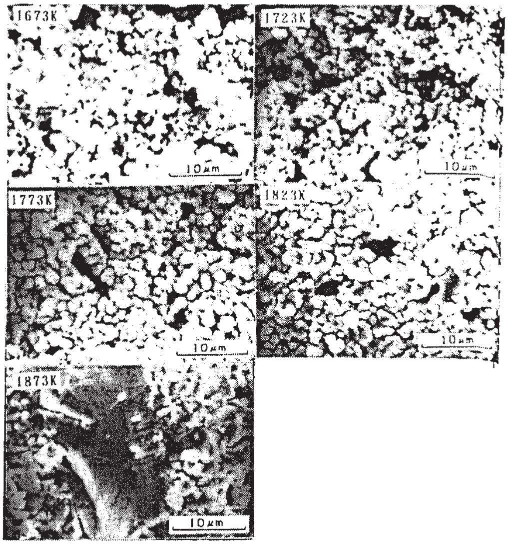

Fig. 9 Microphotograph of bending fracture surface of product fired at indicated temperatures.

5. Conclusions

There have been very few papers which have described an effective use of aluminum sludge as industrial material. With the intention of finding a valuable use, we suggested the application of sludge in the ceramic injection molding. Sludge transformed into α alumina has been investigated as to the possibility of being used as a fine ceramic material for injection molding. As a result, we were able to accomplish the following:

(1)From the analysis of variance using design of experiments, the factor which affected the bending strength of the product most was specified as firing temperature, and the second factor was cylinder temperature.

(2)As the burning temperature rises, the bending strength of the product increases. This is due to the pores reducing inside the product by the smooth sintering at high temperature.

(3)During the confirmation experiment using the optimum condition obtained through SN ratio analysis, the bending strength of the product showed a steady 195.3 MPa. Therefore, it became obvious that the application of α sludge is sufficiently feasible as a ceramics material for injection molding.

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