Product Quality Prediction for Kneading Process in Ceramic Injection Molding*

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The injection molding of ceramics consists of four elementary processes. This paper focuses on the first process, the kneading process of mixing ceramics and organic materials. In this study, current-time curves obtained by a kneader are analyzed from the viewpoint of variance and mean, and it is demonstrated that the quality of sintered products is predictable through these analyses. The method of parameter design of quality engineering is adopted. The variation of sintered products produced after optimizing the molding process on the basis of kneader current measurements, and that of sintered products produced after optimization based on dimension data agree well. These results indicate that prediction of the variability of sintered products at the kneading stage is possible.

Key Words: Ceramics Injection Molding, Kneading Process, Quality Engineering, Control Factors, Orthogonal Array, Optimization, SN Ratio, Production System

1. Introduction

Ceramic injection molding is widely used because three-dimensional shapes can be produced relatively easily and efficiently. Injection molding consists of four elementary processes: kneading, molding, degreasing and sintering. There are reports on process optimization and improvement of the quality of final products; however, satisfactory results have not been obtained. These unsatisfactory results arise from the problems inherent in sequential batch systems; namely, the quality of each process is affected by the preceding process. Accordingly, in the construction of high-quality production systems which include a batch system, it is beneficial to define the major factors in each process and to conduct experiments on the integrated system. In this study, as a means of predicting product quality at an early stage during production, attention is given to the kneading process

which produces the material to be molded, and we propose a method for predicting the quality of the final sintered products from the information obtained during kneading.

2. Experimental Design

2.1 Approach to experiment

In the kneading process, the following items are important: (1) selection of ceramic powder and organic materials, (2) evaluation of kneading conditions, (3) determination of kneader operating conditions, and (4) development of new kneaders.

Items (1) through (3) will be studied in this paper. Characteristics measured in the kneading process include kneader temperature, torque, current and pressure. Kneader current is selected as the characteristic to be analyzed in this study, because it reflects kneading torque and its handling is easy. The parameter design method of quality engineering is applied to optimization of the process.

2.2 Control factors

Control factors are considered for selecting the optimum levels. Control factors are derived from the principle conditions of the four elementary processes.

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Table 1 Control factor and levels experiment

Factors	Level	1	2	3	Unit
A: Wax		2.96	4.07	5.18	wt%
B: Plasticizer		3.70	2.22	0.74	wt%
C: Coupling Agent		0	1.40	0.59	wt%
D: Lubricant		2.22	2.96	3.70	wt%
E: Kneading Time		10	15	20	min
F: Kneading Temperature		423	438	468	K
G: Kneading Rotation Speed		0.72	0.98	1.3	s ⁻¹
H: Mold Temperature		303	313	323	K
I: Molding Temperature		423	408	393	K
J: Injection Pressure		24	40	56	MPa
K: Degreasing Pattern		Low	Mid	High	
L: Sintering Pattern		High	Mid	Low	
O: Binder		5.92	4.07	2.22	wt%

Table 2 Experimental design layout for parameter design

(Orthogonal Array L ₃₆)															wt%
No.	Factor	A	B	C	D	E	F	G	H	I	J	K	L	O	
1		1	1	1	1	1	1	1	1	1	1	1	1	1	15.88
2		2	2	2	2	2	2	2	2	2	2	2	2	2	15.85
3		3	3	3	3	3	3	3	3	3	3	3	3	3	14.66
4		1	1	1	2	2	2	2	3	3	3	3	3	1	14.81
5		2	2	2	3	3	3	3	1	1	1	1	1	1	15.85
6		3	3	3	1	1	1	1	2	2	2	2	2	1	14.66
7		1	1	2	3	1	2	3	3	1	2	2	3	1	17.70
31		1	3	3	3	2	3	2	2	1	2	1	1	3	10.22
32		2	1	1	1	3	1	3	3	2	3	2	2	3	11.48
33		3	2	2	2	1	2	3	1	2	2	3	1	3	12.52
34		1	3	1	2	3	2	3	1	2	2	3	1	3	8.88
35		2	1	2	3	1	3	1	2	3	3	1	2	3	14.37
36		3	2	3	1	2	1	2	3	1	1	2	3	3	10.96

The figures in the table represent the level of each factor

They are listed in Table 1. Kneading time, temperature and number of kneading rotations are selected for the kneading process. An aluminum-group coupling agent is used as a surface processing agent for moldreleasing effects during molding, stearic acid as a lubricant, EVA (ethylene vinyl acetate copolymer) as a binder for the improvement of fluidity and DBP (dibutyl phthalate) as an agent for adding plasticizer by mixing with the material.

These control factors are assigned in an orthogonal array L₃₆, as shown in Table 2. The weight percents of the assist agent are also shown in the right-hand column in Table 2. The reason for the use of the orthogonal array is that if all combinations of control factors were to be tested, the number of experiments would be astronomical; the orthogonal array can reduce the number of experiments and still satisfy all intended purposes effectively.

2.3 Noise factors

Noise factors are the source of variability of the system. The noise factor is derived from the kneading conditions, because the kneading process is considered

to be the major process for determining the product quality. Average grain size of alumina is taken as a noise factor. A material consisting of alumina whose average diameter is 0.8 μm is selected as the first-level N₁, and a mixture of 90% of total alumina consisting of 0.8 μm grains and the rest consisting of 0.4 μm grains, as the second-level N₂. It simulates the fact that the grain distribution varies depending on lot or manufacturer. Even if the grain distribution varies between lots, the condition where uniform products are produced is evaluated as being optimum.

3. Experimental Method: Measurement of Kneading Characteristics

The kneading machine used is a constant-pressure type, where pressure of 0.3 MPa is applied by means of an air cylinder. In the preparation of a mixture, organic materials are measured and poured into a large butt, and then mixed. Use of an auxiliary mixer allows mixing to a certain level followed by the addition of wax to the butt. When the kneader reaches an appropriate temperature, the alumina and organic materials are then poured into the kneaders. The kneading current, which represents the resistive force of the mixture, is recorded during kneading. Kneader pressure is applied during certain periods of the mixing process. An analog output of the kneading current is recorded on a recorder sheet and the recorded graph is converted to coordinate values by a digitizer. These values are compiled in a computer as digital data and processed.

4. Data Analysis

4.1 SN ratio and sensitivity

Variability and magnitude of the kneading current at each sampling time R can be calculated from SN ratio and sensitivity, respectively. They are given by the following equations:

SN ratio

$$\eta = 10 \log \frac{\frac{1}{n}(S_m - V_e)}{V_e} \quad (1)$$

sensitivity

$$S = 10 \log \frac{1}{n}(S_m - V_e) \quad (2)$$

where S_m denotes the sum of the squares of the mean, V_e denotes error variance, and n denotes the number of data used in calculating the SN ratio. The SN ratios represent the coefficient of variance, and the sensitivity represents the mean. One SN ratio can be obtained for each row of the orthogonal array.

4.2 Sampling of kneader current

In the measurement of kneader current, sampling intervals are shortened for the sections containing

high variation with respect to time and increased for stable current periods. The sampling interval is 1 s for the first 10 s, 2 s for 11–50 s, and 5 s for 51–100 s. This gives a total of 40 current values for analysis of the kneading process. SN ratio and sensitivity are calculated for each sampling time R . Accordingly, two kneader currents at the noise factor levels N_1 and N_2 are employed to obtain SN ratios and sensitivity in Eq. (1) and Eq. (2).

5. Experimental Results and Discussion

5.1 SN ratio: Variability of kneading current

The results of the analysis of variance on SN ratio of the orthogonal array L_{36} are shown in Table 3. The first frame represents the variances arising from the main effect of the control factors on the kneading current. The second frame shows the interaction between the control factors and time R , that is, the difference in the variation of the current curve with respect to time changes due to kneading conditions. Figure 1 shows the main effect of significant control factors on SN ratio: the rotation number for kneading, binding agent, wax and plasticizer. With

Table 3 Analysis of variance in SN ratio

Source	f	S(db) ²	V(db) ²
A Wax	2	3685	1842
B Plasticizer	2	3750	1875
C Coupling Agent	2	1357	678
D Lubricant	2	3092	1546
E Kneading Time	2	2063	1031
F Kneading Temperature	2	1988	994
G Kneading rotation speed	2	7619	3809
H Binder	2	4252	2126
e1 Primary Error	19	13181	693
e1' (o-mark Pooled)	23	16507	717
Kneading Time	39	844	21
x x	78	3105	39
x x	78	1248	16
x x	78	1212	15
x x	78	3120	40
x x	78	1633	20
x x	78	1381	17
x x	78	1281	16
x x	78	1071	13
e2 Secondary Error	741	15879	21
e2' (o-mark Pooled)	1209	23508	19
Total	1439	71548	

f: Degrees of freedom S: Sum of squares V: Variance

Fig. 1 SN ratio for each level of controllable factors

the organic materials, SN ratio tends to increase as the amount of plasticizer decreases. Accordingly, the smaller the amount of plasticizer, the greater the curve stability. Also, the SN ratio is higher with a smaller amount of wax and binder; however, the behavior is dependent on the level of each factor and is therefore complex. In the case of the number of rotations of the kneader, the highest SN ratio is obtained at the second level.

The relationships between the kneading time R and the control factors are shown in Fig. 2. At the level of 2.96 wt% of lubricant (Fig. 2(a)), the SN ratio is high on the whole; however it decreases with a decreasing right shoulder profile from the period up to 35 s, and then falls further, but at a slower rate. At the level of 2.22 wt%, the SN ratio decreases rapidly in the period up to 25 s; however, no further change is observed. After 30 s, the SN ratio is similar to that obtained at the level of 5.18 wt%.

In the relationship between R and wax content (Fig. 2(b)), a large variation between levels is observed in the period up to 40 s. The most stable value is obtained at the level of 2.96 wt% of wax; however, the behavior varies in a complex manner. The combination of the levels with higher SN ratio of control factors can be determined as the optimum condition where the variance of the kneader current becomes the smallest. The optimum conditions can be A_1 , B_3 , (C_2) , D_2 , (E_2) , (F_2) , G_2 , O_3 where the numbers in

Fig. 2(a) Relationship between kneading time and lubricant

Fig. 2(b) Relationship between kneading time and wax

parentheses denote the levels with higher SN ratio, but the factor has a small main effect on the SN ratio. The SN ratio in the optimum condition can be estimated from

$$\bar{\mu} = A_1 + B_3 + D_2 + G_2 + O_3 - 4T = 19.23 \text{ (db)} \quad (3)$$

where T denotes the total average of SN ratios. In the same manner, the SN ratio in present conditions can be obtained.

$$\bar{\mu} = A_2 + B_2 + D_2 + G_2 + O_2 - 4T = 9.07 \text{ (db)} \quad (4)$$

The difference in the SN ratio between the optimum conditions and the present conditions is found to be 10.16 db. The gain of 10.16 db means that variance of kneading current in optimum conditions can decrease by 1/3.22 times that under the present conditions. Figure 3 shows the kneader curves under the optimum mixing conditions.

5.2 Sensitivity of the magnitude of kneading current

Table 4 shows the results of the analysis of variance on the sensitivity S obtained by Eq. (2) in

Table 4 Analysis of variance in SN ratio

Source	f	S(db) ²	V(db) ²
A Wax	2	215.93	107.96
B Plasticizer	2	502.49	251.24
C Coupling Agent	2	74.37	37.18
D Lubricant	2	195.83	97.91
E Kneading Time	2	6.33	3.16
F Kneading Temperature	2	142.89	71.44
G Kneading Rotation Speed	2	1780.87	890.43
O Binder	2	34.13	17.06
e1 Primary Error	19	968.00	50.95
e1' (o-mark Pooled)	29	1387.47	47.84
R mixing time	39	961.42	24.65
A×R	78	53.42	0.68
B×R	78	261.08	3.34
C×R	78	71.73	0.91
D×R	78	144.02	1.84
E×R	78	62.67	0.80
F×R	78	74.01	0.94
G×R	78	73.29	0.93
O×R	78	29.95	0.38
e ₂ Secondary Error	741	982.22	1.32
e ₂ ' (o-mark Pooled)	1287	1491.37	1.16
T Total	1439	6634.79	

f: Degrees of freedom S: Sum of squares V: Variance

orthogonal array L_{36} . Sensitivity S represents the magnitude of kneader current. Among the significant factors, those arising from the kneader include the rotation number of the kneader, and those related to organic materials include the plasticizer and wax. In the interaction between the kneading time R and the control factors, effects of the plasticizer and lubricant are significant. The kneading resistance is changed due to the friction and adhesion between powder (powder and grain) and lumps (grain and lump). When all materials become lumps, the current reaches a peak. After this peak, the current suddenly drops. When kneading is sufficient, the current reaches a peak, the intergrain gaps become small, and capillary phenomena develop with the binder as an efficient wetting agent; then the current drops.

Figure 4 shows the effect of the relationship between kneading time and control factors on sensitivity. No difference in sensitivity is observed for levels of plasticizer for the first period up to 15 s; however, some differences are noted afterwards. In the case of plasticity, the sensitivity is high at the level of 0.74 wt%, which means the current is large and the wettability is poor. This phenomenon continues even when mixing time is extended. On the other hand, at the level of 3.70 wt%, the sensitivity drops after 15 s from the beginning of mixing. Nevertheless, it becomes stable at 30 s, and no further change is observed.

Fig. 4 Interaction between kneading time and plasticizer (DBP)

Fig. 3 Kneading torque curve under the optimum mixing condition

Fig. 5 Kneading torque curve under the final optimum condition

6. Composition with Molding Function

The intrinsic function of mold manufacturing is transferability; that is, how well the molded product y resembles the original die M . This can be represented by the following equation:

$$y = \beta M \quad (5)$$

where β denotes the proportional coefficient, and $1-\beta$ represents the coefficient of shrinkage. The signal factor, the die dimension M , is intentionally varied to change the product dimensions.

The SN ratio of transferability is given as an index of the performance of molding by the following equation.

SN ratio

$$\eta = 10 \log \frac{\frac{1}{r}(S_\beta - V_e)}{V_e} \quad (6)$$

$$r = r_0 \sum M_i^2 \quad (7)$$

and

$$S_\beta = \frac{(\sum M_i y_i)^2}{r_0 \sum M_i^2} \quad (8)$$

where S_β is sum of squares of the linear effect of the signal factor, r effective divisor, M_i level value of the signal factor and r_0 number of replications when the number.

Bending test pieces of 4 mm × 3 mm (width and length, respectively) are molded under L_{36} conditions. Their dimensions are measured and SN ratios of transferability are obtained. Analysis of variance on the SN ratios of transferability is performed to determine major factors. The optimum conditions from the viewpoint of transferability are obtained as (A_3), (B_2), (C_1), (D_2), (E_3), (F_3), (G_1), H_3 , I_2 , J_3 , (K_3), (L_1), O_3 . The SN ratio in the optimum conditions can be estimated as $\eta = -25.78$ db ($\sigma = 19.4$ μ m).

The value for present conditions is -36.34 db, and the gain is 10.50 db; Fig. 5 shows the kneading curve for these optimum conditions. This curve is very similar to that of optimum conditions estimated by

the analysis of the kneading current. The SN ratio of transferability by products using material made under the optimum conditions estimated by kneading agreed well with that by products produced under the optimum conditions estimated by the transferability SN ratio.

7. Conclusions

Optimization of the process was carried out on the basis of quality engineering by the use of the kneader current values in the kneading process, which is an early-stage process in ceramic molding. The feasibility of predicting the variability of the dimensions of final sintered products was studied, and the following points were clarified.

(1) The optimization of the kneading process was carried out based on the analysis of kneading current curves in the experiment with major factors selected from the production process.

(2) In stabilizing the kneading curves, optimization of the number of kneader rotations, binder, plasticizer and wax is essential⁽³⁾.

(3) The kneading current curve under the optimum conditions estimated from sintered product dimension measurements agrees well with the curve obtained in (1).

The practical adjustment procedure for the development of an optimum kneading curve and selection of organic materials will be dealt with in the future.

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