Regulation of the Flexural Strength and Porosity of Metakaolin Ceramic Filter Fabricated by Slurry Micro-Extrusion Direct Forming Method

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Abstract: Ceramic filters were widely applied in casting. However, regulating their porosity and strength has been a research difficulty. In this work, high-porosity and high-strength metakaolin-based ceramics were fabricated using the slurry micro-extrusion direct forming method via optimizing four key process parameters, including nozzle internal diameter, height to diameter ratio, filling rate and printing speed. The orthogonal experiments were used to adjust flexural strength and porosity, and the optimal process parameters were obtained by comprehensive scoring and range analysis methods. The predictive models of strengthparameters, porosityparameters and strength-porosity were established and validated. The results showed that the optimal process parameters for high-strength and high-porosity ceramics were 0.51 mm nozzle internal diameter, 60% height-to-diameter ratio, 60% filling rate, and 25 mm/s printing speed. The flexural strength and porosity of the ceramic samples fabricated by optimal process parameters were 17.12 MPa and 48.65%, respectively. The correctness and predictability of three predictive models were proved by two methods, which were the mutual validation and comparison between theoretical and actual values. And the error rates between theoretical and actual results were less than 7%. This work provides guidance for the rapid fabrication of ceramics with adjustable strength and porosity by material extrusion, and the established predictive models can pave the way for its wider application in practice.

Keywords: Material extrusion; Metakaolin-based porous ceramic; Flexural strength; Porosity; Predictive model

1 Introduction

Porous ceramics are widely used in the filtration of molten metal or particulate exhaust gases, catalyst carriers, and gas sensors due to high specific surface area, low bulk density, high-temperature resistance, and corrosion resistance [1]. Traditional processes for preparing porous ceramics include freeze casting and direct foaming [2,3]. However, the traditional processes in the preparation of porous ceramics with complex structures require complex shape molds, and there are some problems such as long manufacturing cycles and low resource utilization. Slurry micro-extrusion direct forming technology, also named direct ink writing (DIW), is flexible in form, simple in operation, and adjustable in pore size, which is suitable for preparing porous ceramics [4]. In practical applications, there are also certain requirements for the strength of porous ceramics in fields such as filtration, catalysis, and insulation. However, there are fewer studies on the correlation between porosity and flexural strength of porous ceramics by DIW, and there is a contradictory relationship between porosity and strength. Therefore, it is important to investigate the correlation between porosity and flexural strength of porous ceramic samples.

2 Experimental procedure

The raw materials used in this study were metakaolin and binder PVA. In the previous experimental research [5], it was found that the four parameters of nozzle internal diameter, height-to-diameter ratio (printing layer height/nozzle internal diameter), filling rate, and printing speed had important impacts on the formability of the green bodies and the property of sintered ceramic, as shown in Fig. 4. Three superior levels of each parameter to well form green bodies were selected. In this work, the four-factor and three-level orthogonal experiments were designed, as shown in Table 1. Comprehensive scoring and range analysis methods were used to study the influence mechanism of each parameter on the flexural strength and porosity of ceramics, and a set of process parameters for fabricating high-strength, high-porosity ceramics were gained [5,6].

Table 1 The orthogonal experimental design

Level	Factors			
	A: Nozzle internal diameter (mm)	B: Height to diameter ratio (%)	C: Filling rate (%)	D: Printing speed (mm/s)
1	0.41	50	60	15
2	0.51	60	80	20
3	0.61	70	100	25

3 Result and discussion

1. Parameter optimization

The results of the orthogonal experiments are shown in Table 2. The optimal process parameters of 0.51 mm nozzle internal diameter, 70% height-to-diameter ratio, 100% filling rate, and 15 mm/s printing speed were obtained by comprehensive scoring and range analysis methods.

Table 2 Results of the orthogonal experiments

Num	Factors				Flexural strength (MPa)	Porosity (%)	Bulk density (g/cm^3)
	A	B	C	D			
1	0.41	50	60	15	24.37±1.48	37.54±2.87	1.64±0.06
2	0.41	60	80	20	23.60±1.54	38.68±3.31	1.61±0.07
3	0.41	70	100	25	31.62±4.34	32.96±0.78	1.71±0.03
4	0.51	60	60	25	16.76±0.74	50.76±5.05	1.28±0.14
5	0.51	70	80	15	26.42±2.62	36.84±2.95	1.66±0.08
6	0.51	50	100	20	32.76±2.38	32.04±1.16	1.73±0.03
7	0.61	70	60	20	16.88±1.17	48.88±2.46	1.36±0.07
8	0.61	50	80	25	21.47±1.06	41.82±1.90	1.53±0.04
9	0.61	60	100	15	30.42±1.45	34.68±1.88	1.68±0.02

The actual flexural strength and porosity of the ceramics fabricated using the optimal parameters were 17.12 MPa and 48.65%, respectively.

2. Predictive models of strength and porosity

Based on the results of orthogonal experiments, Origin software was used to fit multiple linear regression models for strength-parameter, porosity-parameter, and strength-porosity respectively, the fitting results are shown in Eqs. (1), (2) and (3).

$$\sigma=-18.03A-0.06B+0.31C-0.39D+20.85 \quad (1)$$

$$\varphi=27.00A+0.12B-0.31C+0.55D+32.32 \quad (2)$$

$$\sigma = 114.78771e^{-0.03956\varphi} \quad (3)$$

where σ and φ are flexural strength and porosity, respectively

The validation process for Eqs. (1), (2) and (3) can be carried out in two ways. The one approach is the mutual validation between the three models. The other approach is to compare the actual and theoretical values. The specific validation logic is shown in Fig. 1.

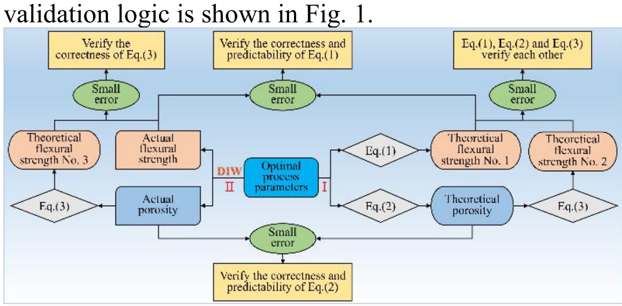

Fig. 1. Validation logic diagram for the fitted models.

3 Conclusion

This work focused on the effect and optimization of four key DIW process parameters on the flexural strength and porosity of metakaolin-based porous ceramics. The orthogonal experiments were designed to investigate the influence law of process parameters on the flexural strength and porosity, and the optimal process parameters were obtained. Models were used to fit the results of orthogonal experiments for achieving the goal of predicting flexural strength and porosity through process parameters. The specific conclusions are drawn as follows:

(1) There was a gradual caramization of metakaolin-based green bodies during the sintering process. The powder particles were tightly bonded to each other, resulting in a decrease in porosity and dimension, and an increase in the strength of the ceramic samples. The volatilization of the organic binder PVA created a large number of pores in the ceramic matrix, which slowed down the reduction of porosity.

(2) The optimal process parameters of 0.51 mm nozzle internal diameter, 70% height to diameter rate, 100% filling rate, and 15 mm/s printing speed for fabricating high-strength and high-porosity ceramics were determined by the comprehensive scoring and range analysis method. Among the process parameters, the filling rate had the most significant effect in changing the flexural strength and porosity.

(3) Predictive models for strength-parameter, porosity-parameter, and strength-porosity were established based on the orthogonal experimental results. The correctness and predictability of the predictive models were proved by two methods of mutual validation among the three models and comparison between the theoretical and actual values. The error rates between the theoretical and actual values did not exceed 7%.

Acknowledgments

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