# Effect of Sintering Temperature on the Precision and Properties of DIW 3Dprinted Porous ZrO<sub>2</sub> Ceramic Filter for Casting

Yuxiao Lin<sup>1, 2</sup>, Yi Wang<sup>1</sup>, Miao Wang<sup>1</sup>, Liang Zhang<sup>1</sup>, Hao Liu<sup>1, 2</sup>, Fuchu Liu<sup>1, 2\*</sup>

School of Mechanical Engineering and Electronic Information, China University of Geosciences, Wuhan 430074, China
 Shenzhen Research Institute, China University of Geosciences, Shenzhen 518057, China
 \*Corresponding address: e-mail: liufuchu@cug.edu.cn (Fuchu Liu)

Abstract: In this manuscrip, porous ZrO<sub>2</sub> ceramic filters were fabricated by direct ink writing (DIW) 3D printing technology, and the effects of sintering temperature on the sintering shrinkage, surface roughness, porosity, flexural strength, and compressive strength of porous ZrO<sub>2</sub> ceramic samples were systematically investigated. Results showed that when the sintering temperature increased from 1400 °C to 1500 °C, the sintering shrinkage of porous ZrO<sub>2</sub> ceramic samples initially increased and then decreased, which can be explained using the variations in ceramic grain size and internal pores caused by different sintering temperatures. When the shrinkage rate was higher, the stepped effect on the side surface of the sintered ceramic samples was smaller, resulting in a higher flatness, and so the surface roughness decreased first and then increased. The main reason for the initial increase followed by a decrease in the flexural and compressive strength of porous ZrO<sub>2</sub> ceramics with increasing sintering temperature was related to the number of grain boundaries and grain boundary bonding properties of the sintered ceramic samples at different sintering temperatures. When the sintering temperature was 1450  $^{\circ}$ C, the sintering shrinkage of porous ZrO<sub>2</sub> ceramics in the X, Y, and Z directions were 8.95%, 8.58%, and 9.21% respectively. The roughness of the side surface was 19.18 µm, the flexural strength was 6.01 MPa, the compressive strength reached 22.70 MPa, the porosity was 62.37%, the bulk density was 2.14 g/cm<sup>3</sup>, and the comprehensive properties were optimal.

**Keywords:** Direct ink writing; Porous ZrO<sub>2</sub> ceramic; Strength; Porosity; Sintering temperature

## **1** Introduction

Porous ZrO<sub>2</sub> ceramics are a high-performance structural material composed of a three-dimensional network skeleton structure, and exhibit advantages such as high specific surface area, low bulk density, excellent high-temperature thermal shock resistance. and high-temperature compressive strength, which are widely used in the field of high-temperature alloy precision casting <sup>[1]</sup>. The use of porous ZrO<sub>2</sub> ceramics for the filtration and purification of molten metals can efficiently remove large and fine suspended inclusions in the metal melt and promote the precipitation of some gases through mechanisms such as mechanical interception, rectification of floating slag, and surface adsorption. This process effectively improves the mechanical properties and service life of high-temperature

alloy precision castings <sup>[2]</sup>. However, traditional methods for preparing porous ceramics, such as direct foaming, gel casting, and freeze casting, have complex preparation processes, long production cycles, require mold development and are difficult to meet the demand for rapid manufacturing of porous ceramics with excellent comprehensive properties such as high porosity and high compressive strength <sup>[3]</sup>. Therefore, there is an urgent need to develop a new method for preparing porous ZrO<sub>2</sub> ceramics with excellent comprehensive properties.

Using DIW 3D printing technology, the high solid content ceramic slurry is deposited on a substrate through an extrusion nozzle, and complex structures are formed by layer-by-layer stacking, effectively enabling the preparation of porous ceramics with pore sizes ranging from micrometers to millimeters.

In this manuscript, DIW 3D printing technology was employed to fabricate porous ZrO<sub>2</sub> ceramic filters using ZrO<sub>2</sub> ceramic powder as the raw material, methyl cellulose (MC) as a binder, and ammonium polyacrylate  $(C_3H_4O_2)$  as a dispersant. On this basis, the influence of different sintering temperatures on the accuracy (sintering shrinkage and surface roughness) and properties (porosity, flexural strength, and compressive strength) of porous ZrO<sub>2</sub> ceramics were investigated. The microstructural evolution of porous ZrO<sub>2</sub> ceramic samples at different sintering temperatures was analyzed using scanning electron microscopy (SEM). Moreover, an attempt was made to establish the relationship between the properties and microstructural evolution of porous ZrO<sub>2</sub> ceramics. Finally, porous ZrO<sub>2</sub> ceramic filters with optimal comprehensive properties were successfully prepared, with a surface roughness of 19.18 µm, porosity of 62.37%, and a compressive strength of 22.70 MPa, at a sintering temperature of 1450 °C.

## 2 Experimental procedure

First, a uniformly dispersed ZrO<sub>2</sub> ceramic slurry was prepared. The composition ratio of the ceramic slurry is listed in Table 1.

Subsequently, porous ceramic green bodies were printed with a self-built DIW 3D printing device. The forming process parameters are listed in Table 2.

Table 1	. Porous	ZrO <sub>2</sub>	ceramic	slurry	com	position.
		_				

Slurry composition	ZrO <sub>2</sub>	2 wt.% MC	C <sub>3</sub> H <sub>4</sub> O <sub>2</sub>
Content (wt.%)	79	20.3	0.7

P	Part 15: 1	Moulding	Material	and	Technol	logy

Table 2. Dive printing parameters	useu in this manuscript
Printing parameter	Parameter value
Nozzle inner diameter (mm)	0.41
Filling rate (%)	50
Layer height (mm)	0.246
Printing speed (mm/min)	700

Table 2. DIW printing parameters used in this manuscript

ting speed (mm/min) Based on the test results from the TG-DSC simultaneous thermal analyzer, the sintering temperature curves for porous ZrO<sub>2</sub> ceramic samples were designed. Initially, the temperature was increased to 500 °C at a rate of 2 °C/min, and held for 2 hours to ensure thorough removal of organic binders and complete evaporation of residual moisture from the samples. Subsequently, the temperature was increased to 800 °C at a rate of 2 °C/min, and held for 1 hour to facilitate the decomposition of internal organic impurities. Then, the temperature was increased at a rate of 2.5 °C/min to 1400 °C, 1450 °C, and 1500 °C, respectively, and maintained for 2 hours at each temperature to promote full migration of ceramic particles and bonding at grain boundaries, enhancing ceramic densification. Finally, the sintered ceramic samples were obtained by furnace cooling to room temperature.

### **3** Result and discussion Microstructural evolution

When sintered at 1400  $^{\circ}$ C, the grain migration distance was small, and grain boundaries bonding was slow, resulting in a loosely structured overall grains arrangement and larger pores. As the temperature further increased to 1450 °C, the larger pores gradually shrank, and the bonding between grain boundaries became tighter. Once the sintering temperature was increased to 1500 °C, the grain migration distance increased, and the grains fused with each other, disrupting grain boundaries bonding and forming larger agglomerates. The microstructural evolution results indicated that high sintering temperatures can lead to abnormal grain growth and weakened grain boundary bonding. Simultaneously, due to the abnormal grain growth, the ceramic samples were prone to microcracks during the sintering process, significantly affecting the ceramics' mechanical properties.

As the sintering temperature increased, the average grain size continuously enlarged. For instance, when the temperature increased from 1400 °C to 1450 °C, the average grain size coarsened from  $1.57 \pm 0.08 \ \mu m$  to  $1.91 \pm 0.12 \ \mu m$ , indicating a pronounced trend of grain growth. As the temperature further increased to 1500 °C, the average grain size continued to coarsen, due to the abnormal growth of some grains, although the increasing trend decelerated, as depicted in Fig. 1.

## **Sintering property**

The shrinkage rate, surface roughness, porosity, density, flexural and compressive strength of porous  $ZrO_2$  ceramic samples at different sintering temperatures are shown in Table 3.



 
 Table 3. Effect of different sintering temperatures on the precision and properties of porous ZrO<sub>2</sub> ceramics

Sintering temperature	Flexural strength	Compressiv e strength	Porosi	Density	Roughn ess	Sh	rinka (%)	ge
(°C)	(MPa)	(MPa)	ty (%)	(g/cm°)	(µm)	Х	Υ	Ζ
1400	3.9	18.3	66.8	1.9	29.3	7.8	6.5	9.1
1450	6.1	22.7	62.4	2.1	19.2	8.9	8.6	9.2
1500	3.6	17.8	64.3	2.1	26.7	7.9	6.6	9.1

## 4 Conclusion

(1) The average grain size of porous ZrO<sub>2</sub> ceramics continuously increases with elevated sintering temperature, but the increasing trend gradually slowes down.

(2) The sintering shrinkage rate of porous ZrO2 ceramics initially increases and then decreases with elevated sintering temperature. When the shrinkage was higher, the side surface of the ceramic exhibited higher smoothness, hence the surface roughness decreased initially and then increased with elevated sintering temperature.

(3) Due to the higher number of grain boundaries and higher bulk density of porous  $ZrO_2$  ceramics sintered at 1450 °C, the flexural and compressive strength of the ceramic samples were higher.

## Acknowledgments

This work was supported by Guangdong Basic and Applied Basic Research Foundation [No.2024A1515013258] and Free Exploration Basic Research Project of Local Science and Technology Development Funds Guided by the Central Government [No.2021Szvup158].

## References

- Olson III R A, Martins L C B. Cellular ceramics in metal filtration. J. Advanced Engineering Materials, 2005, 7(4): 187-192.
- [2] Li S, Baeyens J, Dewil R, et al. Advances in rigid porous high temperature filters. J. Renewable and Sustainable Energy Reviews, 2021, 139: 110713.
- [3] Ohji T, Fukushima M. Macro-porous ceramics: processing and properties. J. International Materials Reviews, 2012, 57(2): 115-131.