Micro-Extrusion 3D Forming and Anti-hydration Strengthening of Water-Soluble Calcia Ceramic Cores for Manufacturing Complex Hollow Castings

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Abstract: Poor forming accuracy and susceptibility to hydration swelling were bottlenecks in the casting application of micro-extruded 3D printed CaO ceramic cores. In this manuscript, three key process parameters, namely, the inner diameter of the needle (d), the layer height (h) and the printing speed (v), were optimized through response surface experiments, and the regression equations for surface roughness were established and analysis of variance (ANOVA) was carried out. The results showed that the main factor affecting the forming accuracy of the printed green bodies was the inner diameter of the needle, followed by the layer height, and the printing speed was the least affected. When d=0.41mm, h=0.31 mm, v=29.87 mm/s, the response value was the smallest and the billet indicated the best quality. The moisture absorption rate of CaO ceramic cores was tested by introducing Nano-ZrO₂ powder, and the results showed that when the addition of Nano-ZrO₂ was 10 wt.%, the moisture absorption rate of CaO ceramic cores decreased by 43.9%, and at the same time the shrinkage rate decreased by 21.5%, the flexural strength increased by 63.8%, and the water solubility rate in 60 °C water was 4.21 g/s·m², which was important for the rapid moldless. The SEM microscopic morphology and XRD physical phase analysis showed that the introduction of the additive generated a new phase substance CaZrO₃, which was uniformly distributed around the CaO experience to effectively reduced the moisture absorption rate of CaO. The research results were of great theoretical and practical significance for the rapid moldless accurate forming of hydration water-soluble CaO ceramic cores.

Keywords: CaO ceramic core,Extrusion 3D printing technology,Formability,Hydration resistance

1 Introduction

The structure of new generation aerospace, high-speed rail and new energy vehicle castings develops in the direction of complexity and integrality, the casting cores became very complex, and it is difficult to clean the inner cavity of the casting, the traditional core preparation process needed to form a number of small cores through the mold, and then obtained the whole core from the group, which was a complex process, high cost, and low precision^[1,2]. The preparation of water-soluble CaO ceramic cores by microextrusion 3D printing technology can simultaneously solved the forming and cleaning problems of complex cores. However, the disadvantages of CaO ceramic cores, such as reduced strength due to moisture absorption and low extrusion precision, had seriously hindered their practical application^[3]. In this manuscript, a three-factor, three-level Box-Behnken test was firstly conducted based on the response surface method with the inner diameter of the needle, layer height/inner diameter and printing speed. A quadratic regression model was established using the surface roughness of the printed ceramic green body, and the relationship between the three factors and the response value surface roughness was discussed by analysis of variance (ANOVA), and the interaction between the three factors was analysed to determine the optimal process parameter combinations. Then, additives were introduced to improve the anti-hydration properties of CaO, and the effects of different Nano-ZrO2 additions on the linear shrinkage, flexural strength, moisture absorption and water dissolution rate of ceramic cores were investigated. The microstructural evolution of CaO samples containing different amounts of Nano-ZrO2 additives was described by SEM microscopic morphology analysis and XRD physical phase analysis, and the optimal amount of Nano-ZrO₂ was determined based on the comprehensive evaluation of the anti-hydration and water solubility rate, which realized the unity of the anti-hydration and water solubility rate of the CaO ceramic cores. This study provided a new way to obtain high-performance, high-precision, anti-hydration water-soluble ceramic cores, which was of great theoretical and practical significance for industrial development.

2 Experimental procedure

1250 mesh CaCO₃ powder was used as the matrix material and Nano-ZrO₂ powder with an average particle size of 30 nm was used as the additive. A 30 wt.% solution of polyethylene glycol (Mw=4000 g/mol) was configured as a binder using deionised water as a solvent. Ceramic green bodies were micro-extruded and formed using an indigenously built printing device. Printing parameters were controlled and adjusted using Simplify 3D software. Green bodies printed first print substrate on the completion of natural drying and then transferred to a constant temperature drying oven to remove excess moisture on the surface. The fully dried green bodies were transferred to a high temperature sintering furnace, and the CaO ceramic cores are obtained after stepwise sintering to 1400°C. The CaO ceramic cores were then sintered at a constant temperature to remove excess moisture from the surface.

3 Result and discussion

According to the Box-Behnken design principle, a threefactor, three-level response surface experimental table was designed using the inner diameter of the needle (A), layer height/inner diameter (B) and printing speed (C) as the independent variables, and the surface roughness of the ceramic core green body as the response value Y.

By measuring the surface roughness of green bodies printed with different parameters, the experimental results were subjected to quadratic regression analysis to establish the regression equation for the surface roughness of ceramic core green bodies:

$$Y = 55.05 + 12.42A + 4.57B - 2.32C +$$

2.01*AB* + 0.69*AC* - 2.94*BC* + (1)
3.16*A*² - 3.35*B*² - 4.75*C*²

After analysis of variance, factors A, B and C had a significant effect on the surface roughness of the green body in the order of A > B > C. The optimal parameter combination was obtained by analysing and solving the regression equation, and the surface roughness of the ceramic core green body was 35.41 µm when the A was 0.41 mm, the B was 0.75, and the C was 29.87 mm/s.

Due to the fact that CaO was highly susceptible to hydration expansion and pulverisation during storage, its mechanical properties and dimensional accuracy were affected. Therefore, its hygroscopic resistance was improved by introducing additives, and five groups of pastes were formulated with Nano-ZrO₂ content from 0-20% at 5% intervals. After micro-extrusion 3D printing, drying, and sintering, CaO ceramic cores were obtained and tested for performance, and the results showed that the moisture absorption rate of all five groups of ceramic cores increased with the increase of storage time and decreased with the increase of Nano-ZrO2 content. In addition, the linear shrinkage of the ceramic cores decreased with the increase of the Nano-ZrO₂ content, while the bending strength showed a gradual increase. As can be seen from the XRD patterns, the diffraction peaks of the ceramic cores with the addition of Nano-ZrO₂ generate CaZrO₃ phase. From the SEM microscopic morphology, it can be seen that the CaO grains were surrounded by the formed CaZrO₃, which effectively inhibits the moisture absorption. The water solubility rate of the moisture-resistant reinforced cores was tested by placing them in different water temperatures, and the results showed that the water solubility could still be faster at water temperatures ranging from 60°C to 80°C. In addition, the higher residual temperature during the water-soluble cleaning creates a good condition for the rapid removal of the ceramic core.



4 Conclusion

In this manuscript, the process parameters of microextrusion 3D printing forming ceramic cores were optimized by response surface experiments, and the best surface quality of ceramic core green bodies with a roughness of 35.41 μ m was achieved when the inner diameter of the needle was 0.41 mm, the layer height/inner diameter ratio was 0.75, and the printing speed was 29.87 mm/s. When the amount of NanoZrO₂ was added at 10%, the hygroscopicity of the ceramic cores with CaO decreased by 43.9%, which was due to the generation of hydrophobic phase CaZrO₃ after the introduction of the additive.

Acknowledgments

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

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