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THE PREPARATION OF CALCIA-BASED CERAMIC SLURRY FOR RAPID MANUFACTURING HOLLOW TURBINE BLADE BASED ON STEREOLITHOGRAPHY

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ABSTRACT: A new water-soluble calcia-based integral ceramic mold was developed for hollow turbine blade by combining stereolithography and gel casting. The influences of solid loading, and grain gradation on the rheological property of calcium carbonate ceramic slurry was investigated. For the more, the effect of zirconia additions on the hydration resistance of calcia-based ceramic mold was studied. The results show that the calcium carbonate ceramic slurry with 60 vol % solid loading and 0.83 Pa·s viscosity was obtained by three graded grain gradation with 40μm:5μm:2μm=56:5:39. The drying shrinkage of green body is less than 0.08% by vacuum freeze drying and zirconia can improve the hydration resistance of calcia-based ceramic mold significantly. The slurry of calcium carbonate ceramic can be used for manufacture the integral calcia-based ceramic mold for turbine blade and the calcia-based integral ceramic mold fabricated by this method can overcome the poor leachability of Al₂O₃ or SiO₂ based ceramic molds and decrease the production costs.

INTRODUCTION

The hollow turbine blade is the most critical component of aircraft engine and marine power equipment. High quality ceramic mold with complex interior shapes, good uniformity, excellent surface finish, and precise cavities is the basis of investment cast hollow turbine blade (Wu et al., 2009; Lu et al., 2013). With the development of aerospace industry, the structures of turbine blade are becoming more complex. In the traditional fabrication process of ceramic mold, the ceramic cores and shell are fabricated separately and then assembled together by core prints (Wu et al., 2009). But this method has some shortcomings, such as assembly errors, time consuming and expensive for new product development.

The integral ceramic mold fabricated by gel-casting and stereolithography (SL) provides a new method for fabricating hollow turbine blade (Wu et al., 2010). The common ceramic molds or cores are usually made of fused silica or alumina due to its excellent properties, such as low density, good high-temperature resistance, good high-temperature strength and good thermal shock resistance (Hurwitz et al., 2010; Huang et al., 2004). However, the fused silica or alumina are difficult to remove, chemical corrosion or mechanical stripping methods are always used to remove the fused silica or alumina cores, which need dedicated device or etch castings (Ding et al., 2011; Wang et al., 2013). What’s more, this process makes the castings become more expensive and low efficient production. A new kind of cores or molds with good comprehensive property and good leachability is required. The calcia-based ceramic cores or molds have been considered as a candidate due to its advantages, such as low cost, worldwide abundance of raw material, high melting temperature, excellent thermodynamic stability and easy to remove (Liu et al., 2015). The calcium oxide can be easily obtained by the decomposition of calcium carbonate at 1273 K, and the calcium carbonate is insoluble in water, which can apply to aqueous gel-casting. Therefore,

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CaCO₃ is reasonably used as raw material for manufacturing calcia-based ceramic mold.

Gel-casting is a novel ceramics forming technology for fabricating high quality and complicated shape ceramic products, which was invented by Oak Ridge National Laboratory in the USA (Omatete et al., 1997). In the gel-casting process, the key factor is the preparation of ceramic slurry. The ceramic slurry with high solid loading, low viscosity and good flowability is directly affect the quality of gel-casting.

In this paper, a ceramic slurry of calcium carbonate with 60 vol % solid loading and 0.83Pa·s viscosity was prepared by grain gradation. The influences of grain gradation and solid loading on the rheological property of calcium carbonate ceramic slurry was investigated. Furthermore, the effect of zirconia additions on the hydration resistance of calcia-based ceramic mold was studied.

EXPERIMENTAL PROCEDURE

Experimental materials
Commercially available calcium carbonate powder (D₅₀=40 μm, 5 μm, 2 μm, supplied by Shenzhen Leadfu New Material Co. LTD, Shenzhen, China) was used as the raw materials. Its chemical composition is shown in Table 1. Zirconia powder (D₅₀=5 μm, supplied by Sinopharm Chemical Reagent Co, Ltd, Shanghai, China) was used as the additives.

| Chemical composition of calcium carbonate powder (wt %) |
|----------------|----------------|----------------|----------------|----------------|----------------|
| CaCO₃           | Fe₂O₃          | SiO₂           | MgO            | SO₄²⁻         | Other carbonates |
| ≥98.8           | ≤0.02          | ≤0.01          | ≤0.01          | ≤0.01          | ≤0.05           |

Experimental procedure
Acrylamide (CH₂CONH₂, AM, supplied by Tianjin Kemiou Chemical Reagent Co, Ltd, Tianjin, China) and N,N-methylene diacrylamide (C₇H₁₀N₂O₂, MBAM, supplied by Tianjin Kemiou Chemical Reagent Co, Ltd) were used as organic monomer and cross-linking agent, respectively, which were dissolved (24:1) in deionized water. Sodium polyacrylate (PAAS, supplied by Sinopharm Chemical Reagent Co, Ltd, 2.5 wt% of solid powders) was then added as the dispersant. Then a premixed solution with 15% concentration was prepared by magnetic stirring for 3 h. The calcium carbonate powder with the appropriate amount was added into the premixed solution using mechanical stirring. Finally, the ceramic slurry was obtained after ball milling for 40 minutes. The ceramic slurry was poured into resin mold which was fabricated by SPS600B Rapid Prototyping Machine (Shaanxi Hengtong Intelligent Machine Co., Ltd, Xi’an, China) using photosensitive resin (SPR 8981; Zheng bang Ltd., Zhuhai, China) under vacuum and vibration conditions. At last, the green body was put into a furnace, heated to 1500 °C and kept for 3 hours after freeze-drying for 48 h.

Measurements
The viscosity of ceramic slurry was measured by SNB-2 Rotary Viscosimeter (Nirun Intelligent Technology Co., Ltd, Shanghai, China) with a rotating speed of 30r/min. The filling ability of ceramic slurry was investigated by detected by the micro X-ray imaging system (Y.Cheetah; YXLON, Hamburg, Germany) with a scanning resolution of about 45 μm. The drying shrinkage of
green body was calculated according to Eq. (1), with the green ceramic samples of a nominal size of 4 mm × 10 mm × 60 mm.

\[ \varepsilon = \frac{L_1 - L_2}{L_1} \times 100\% \] (1)

Where \( \varepsilon \) the shrinkage of sample, \( L_1 \) is the length of sample before drying, \( L_2 \) is the length of sample after drying.

**RESULTS AND DISCUSSION**

**Effect of grain gradation on the viscosity of ceramic slurry**

Grain gradation is a common technique in ceramic slurry preparation, which is different particle size powders mixed in a certain proportion, in order to improve the solid loading and reduce the viscosity of the slurry. Because the small particles can be better filled in the gaps between the larger particles, the particles so as to achieve the closest particle crowding effect by grain gradation. Therefore, particle size distribution of the ceramic slurry can not only obtain a lower viscosity, but also improve the density of ceramic body. Grain gradation can be divided into continuous and discontinuous distribution. In this study, the grain gradation should be seen as a continuous distribution due to the ceramic powder is wide distribution particle, which satisfy the Dinger-Funk equation (Funk et al., 1994).

\[ U_t(D_i) = \frac{D_i^n - D_{\text{min}}^n}{D_{\text{max}}^n - D_{\text{min}}^n} \] (2)

Where \( D_i \) is the particle size, \( U_t(D_i) \) is the cumulative distribution under \( D_i \), \( D_{\text{max}} \) is the maximum particle size, \( D_{\text{min}} \) is the minimum particle size, \( n \) is distribution index and \( n = 0.15 \) in this study.

Figure 1. Fitting curve and D-F curve

In fact, the particle size distribution according to the Dinger-Funk model is the mixed system distribution which contains a variety of particles. Therefore, the grain gradation problem can be converted to the fitting problem between distribution curve of mixed particles \( U_d(D_i) \) and Dinger-Funk equation curve \( U_t(D_i) \). In other word, this problem can be seen as minimizing the error
between actual curve $U_d (D_i)$ and the target curve $U_t (D_i)$. Figure 1 is the fitting curve and D-F curve, which is fitted using least square method by MATLAB. While the constraints is $50\% \leq V_{40} \leq 70\%$; $5\% \leq V_5 \leq 30\%$; $5\% \leq V_2 \leq 50\%$, $V_{40} + V_5 + V_2 = 1$, where $V_i$ is the $i$-th particle volume fraction. Finally, the three graded grain gradation with $40\mu$m:$5\mu$m:$2\mu$m=$56:5:39$ is obtained by this fitting process.

**Effect of solid loading on the viscosity of ceramic slurry**

High solid loading and low viscosity are benefit for producing compact ceramic parts using gel casting. The effect of solid loading on the viscosity was tested, as shown in Figure 2. As can be seen from Figure 2, the viscosity showed exponential growth with the increasing of solid loading. When the solid loading is lower than 60 vol%, the viscosity is very low, but the low solid loading will cause the low density of ceramic body and bad performance of ceramic mold. When the solid loading reaches 60 vol%, the viscosity is 0.83 Pa·s, which can meet the requirement of gel-casting. However, when the solid loading is larger than 60 vol%, the viscosity is >1 Pa·s, which is not fit for gel-casting (Tulliani et al., 2013). Therefore, the solid loading of 60 vol% is the most suitable for gel-casting. Then, some samples were fabricated by using the three graded grain gradation, 60 vol% ceramic slurry, after freeze-drying for 48 hours. The dry shrinkage of green body was calculated and it was less than 0.08%, indicated that the samples has a high density.

![Figure 2. Effect of solid loading on the viscosity of ceramic slurry](image)

**Effect of zirconia on the hydration resistance of calcia-based ceramic mold**

The application of calcia-based ceramic cores or molds have been inhibited due to their drawback of poor hydration resistance. Therefore, improve the hydration resistance is significant for the application of calcia-based ceramic mold. Additions of ZrO$_2$ can improve the hydration resistance of calcia-based ceramic mold effectively. Figure 3 indicates the solubility test results of the watersoluble calcia-based ceramic samples leached in water. We can see that both groups of calcia-based ceramic sample added zirconia or not, were hydrated. The sample without adding zirconia dissolved in water very quickly, just in 10 mins. However, hydration rapidly is not beneficial to transport or store in practical engineering. The sample adding 2 wt% zirconia also dissolved in water in 6 hours, indicated that the hydration resistance of calcia-based ceramic mold was improved. Therefore, the calcia-based ceramic mold adding zirconia is convenient to transport or store, because the hydration is not too fast.
Figure 3. Effect of zirconia on the hydration resistance of calcia-based ceramic samples. (a) Samples without adding zirconia 0 min in water, (b) Samples without adding zirconia 10 min in water, (c) Samples adding 2 wt% zirconia 0 min in water, (d) Samples adding 2 wt% zirconia 6 hour in water

Case study
A calcia-based green body of turbine blade ceramic mold was manufactured by using the CaCO₃ ceramic slurry. The industrial CT images were investigated after freeze-drying, as shown in Figure 4. We can see that the mold was filled completely using the CaCO₃ ceramic slurry prepared in this paper, even the most complex structures, such as U-Shaped structure and the exhaustion edge.

Figure 4. Industrial CT images of calcia-based ceramic mold of turbine blade. (a) U-Shaped structure of ceramic mold; (b) Fine structure of ceramic mold

CONCLUSIONS
In conclusion, the calcium carbonate ceramic slurry with good filling capacity was successfully prepared in this paper, which can be used for manufacturing the integral calcia-based ceramic mold for turbine blade by gel-casting based on stereolithography. The calcium carbonate ceramic slurry with a solid loading of 60 vol % and a viscosity of 0.83 Pa·s was obtained by three graded grain gradation with 40μm:5μm:2μm=56:5:39. The drying shrinkage of green body is less than 0.08 % by vacuum freeze drying. Zirconia can improve the hydration resistance of calcia-based ceramic mold significantly and eliminate the influence of hydration on the application of calcium-based mold. A calcia-based ceramic mold of turbine blade was manufactured by using the calcium carbonate ceramic slurry.

ACKNOWLEDGMENTS

This research was supported by the National Basic Research Program of China (Grant No. 2013CB035703) and the Fundamental Research Funds for the Central Universities. The authors are grateful for the grants.

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