

# Preparation And Degreasing Process Optimization of Light-Curing Slurry for Alumina Ceramics

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**Abstract.** Aluminum oxide ceramic light-curing paste preparation and degreasing has always been an important part of the production and manufacturing of alumina ceramics, and is also the key to ensure the quality of alumina ceramics, so it has been the attention of relevant technical personnel and workers and concerns. The traditional alumina ceramic light-curing paste preparation and degreasing process still has some shortcomings, and if these shortcomings are not compensated in time, the overall quality of alumina ceramics will be affected, so the alumina ceramic light-curing paste preparation and degreasing process must be continuously optimized. In this regard, this paper analyzes in detail the preparation and degreasing process optimization of light-curing slurry for alumina ceramics. The preparation of alumina ceramic light-curing paste with incomplete degreasing and other undesirable phenomena will not only lead to the occurrence of a large number of porosity, but also increase the incidence of defects such as cracks, thus reducing the quality of ceramics. Therefore, as the relevant staff, not only should pay great attention to the preparation and degreasing of alumina ceramic light-curing paste, but also according to the various preparation and degreasing problems that occurred in the past, and constantly optimize the existing alumina ceramic light-curing paste preparation and degreasing process, if necessary, with the help of 3D printing technology, light-curing molding (SLA) technology modern high-tech technology, to constantly optimize the alumina ceramic light-curing paste Preparation and degreasing process

**Keywords:** Alumina ceramics; light-curing paste; preparation and degreasing; light-curing printing; dispersant; process optimization.

## 1. Introduction

"Alumina ceramics (Figure 1)" is essentially a kind of alumina ( $Al_2O_3$ ) as the main ceramic material made of a ceramic. Alumina ( $Al_2O_3$ ) not only has good conductivity, but also has strong strength and high temperature resistance, which determines the use of alumina ( $Al_2O_3$ ) made of ceramics with strong conductivity, mechanical strength and high temperature resistance.



**Figure 1** Alumina ceramics

"Alumina ceramics are also characterized by structural stability, corrosion resistance, wear resistance and high hardness, and are used not only in medical and biological fields and aerospace applications, but also as a key material for artificial teeth, sewage treatment substrates and air compressor blades. Alumina ceramic molding is the key to making alumina ceramics, so it is important to reasonably select additive manufacturing technologies for alumina ceramic molding, and common additive manufacturing technologies include selective laser sintering (SLS), selective laser melting (SIM) and light-curing molding (SLA), among which SLA technology is most widely used [1].

Alumina ceramics can be classified as follow:

(1) High-purity type alumina ceramics. Alumina content of more than 99.9% and high sintering temperature (up to 2000 °C), transmission wavelength standard range values of 1 ~ 6, can be used as raw materials for molten glass to replace the platinum crucible, specific high hardness, high temperature resistance and other advantages.

(2) ordinary alumina ceramics: alumina content range value of 75% to 80%, can be used as raw materials for high-temperature crucible, refractory furnace tube, water valve piece, etc., with strong corrosion resistance and wear resistance and other advantages.

## **2. Alumina ceramics production process and firing technology**

### **2.1. Production process**

#### **2.1.1 Powder preparation**

That is, in accordance with the relevant requirements and standards will be alumina raw material processing molding alumina powder material, alumina powder particle size must be controlled in the range of <math> < 1 \mu\text{m}</math>. It is also necessary to control the purity of alumina and manufacturing temperature according to the type of alumina ceramics, such as the manufacture of high-purity alumina ceramics, whose alumina purity must be controlled within the range of  $\geq 99\%$ . If semi-automatic or fully automatic dry pressing is used, it is also necessary to spray the powder using the spray granulation method to ensure that the powder is in the shape of a round ball.

#### **2.1.2 Ceramic Forming**

Forming methods include dry pressing, injection molding, extrusion, and cold isostatic pressing. The most common methods are dry press molding and slurry molding, as follows: ① Dry press molding: mainly used for alumina ceramic molding with simple shape and inner wall thickness over 1mm, the presses that can be used for dry press molding are hydraulic and mechanical, and semi-automatic or fully automatic molding operations can be performed. The maximum pressure (200Mpa) and frequency (15~50 pieces/min) of the press need to be controlled during the dry press molding process. ② Injection molding method: It is the earliest alumina ceramic molding method, mainly applied to the molding of large size and complex shape ceramics. The injection molding process needs to be applied to the solvent, and water is the medium of the melt. It is necessary to add a certain amount of water in a standard proportion while adding the uncoupling agent and binder to ensure that the ceramics can be fully exhausted after sufficient grinding to accelerate the curing of the slurry, so as to achieve the purpose of molding [1-2].

### **2.2. Sintering technology**

Sintering is essentially the process of densifying granular ceramic blanks and forming a solid material, and effective sintering can not only exclude inter-particle cavities within the blanks, but also effectively exclude small amounts of gases and impurities organic matter to promote inter-particle bonding, thus forming a new substance. Electric furnace is the key equipment of alumina ceramic sintering, is also the most frequently used heating device, in the use of a good control of the rated power of the furnace and constant temperature power, etc., to ensure the safety of electric furnace operation. Sintering can also be divided into atmospheric pressure sintering, pressureless sintering, hot pressure sintering and static pressure sintering, etc. Atmospheric pressure sintering is used most frequently and has the advantages of low cost and simple operation, so many alumina ceramics are sintered using this method [2]. But the method will be affected by the product length, pressure transfer medium, etc., so in the sintering of high growth need to control the length of the product, and according to the actual choice of pressure transfer medium, etc.

### 3. Alumina ceramics sintering conditions and technical specifications

Alumina ceramics need to be sintered professionally in a studio or firing room with large space and suitable temperature, and also need to control the size and power of the propulsion machine, the accuracy of the pyrometer, temperature control points, etc. The relevant specifications and parameters are shown in Table 1.

**Table 1** Sintering conditions of alumina ceramics

Related equipment and index	Specifications and parameters
Work chamber	13720L/280x2W/450H (including push plate)
Push plate	240L/270W/40H/mm
Material	Special grade jade resistant mullite (DGM90)
Rated power Approx	210KW
Constant temperature power Approx	130KW
Rated working temperature in high temperature zone	1400°C
Temperature control points	10
Instrument temperature control accuracy	±2°C (after steady state)
Surface temperature rise of furnace side wall	≤55°C
Propulsion speed	500~1500mm/h
Keep warm time 5h	5h(propulsion speed: 980mm/h)
Main propulsion machine thrust	3T
Working power supply	3-phase 4-wire, 380V
Max. physical size of electric kiln approx	16000L/1800W/1700H mm

Alumina ceramic sintering process also needs to observe a variety of technical indicators, alumina ceramic sintering must be carried out in strict accordance with the relevant technical indicators, such as alumina ceramic content, density and Rockwell hardness and other indicators, see Table 2.

**Table 2** Technical indicators of alumina ceramic sintering

Indicators	Parameters
Alumina ceramic content	≥92%
Density	≥3.6 g/cm <sup>3</sup>
Rockwell hardness	≥80 HRA
Compressive strength	≥850 Mpa
Fracture toughnessKIC	≥4.8MPa·m <sup>1/2</sup>
Flexural strength	≥290MPa
Thermal conductivity	20W/m.K
Thermal expansion coefficient	7.2×10 <sup>-6</sup> m/m.K

### 4. Preparation and degreasing of alumina ceramic light-curing slurry

#### 4.1. Preparation of alumina ceramic light-curing slurry

(1) Dissolve the solid dispersant in ethanol solution using ultrasound, then weigh the alumina powder according to a certain ratio and fuse the alumina powder with the dispersant, and finally pour it into the ethanol solution (the dose is 200 ml).

(2) Grinding treatment. The mixed alumina powder can be poured into the ball mill jar (see Figure 2 for the grinding ball), and then the ball mill is mixed at 300 r/min (time is 3h); the ball mill procedure is: first rotate clockwise for 10 min, then rotate counterclockwise for 10 min, and the interval time of

successive rotations is 5 min; the mixed material is taken out and put into the environment of 60°C for drying, and finally repeated grinding for several times. The modified powder was obtained.



**Figure 2** Alumina ceramic grinding ball

(3) The photosensitive resin HDDA, HEA and TMPTA were weighed according to the volume ratio of 6:3:1, and a certain amount of photoinitiator was added according to the actual; then the mixed substances were fully mixed by magnetic stirring method to obtain the premixed resin; finally, the resin and modified powder were mixed and milled, and the same ball milling method was used to grind, with the ball milling speed of 400 r/min and the ball milling time of 3h. The ball milling time is 3 h, thus obtaining the alumina ceramic light-curing slurry and saving it in the designated storage [3].

#### 4.2. Light-cured printing and molding

After successful preparation of alumina ceramic light-cured slurry, it needs to be molded, and light-cured printing molding is a new molding technology that has emerged in recent years to quickly mold alumina ceramic light-cured slurry. The principle of light-curing printing and molding is to use light-curing printer to quickly build a three-dimensional model, and then pour the prepared alumina ceramic light-curing paste into the model, and finally through the Preform software slicing and printing, so as to complete the alumina ceramic molding. elicitor, but also able to quickly build 3D models, slice and print, improving the molding speed and efficiency [4].

The printing and molding of common alumina ceramic pastes can usually be completed successfully, but the preparation of high solid content, low concentration alumina ceramic pastes requires the setting of reasonable printing parameters, such as the exposure energy of the paste surface, photosensitive parameters, molding layer thickness, and light source intensity, to avoid undesirable phenomena such as photoassimilation reactions due to the absorption of UV light by the photosensitive resin during the light-curing printing process, thus reducing the cured layer thickness. This confirms that the light-curing printing parameters directly affect the cured layer thickness, and this influence process is in accordance with Lambert's law, corresponding to the following expressions.

$$C_d = S_d I_n \left( \frac{E_0}{E_d} \right)$$

Where.

$E_d$  -- the exposure energy irradiated to the surface of the slurry.

$E_0$  --critical exposure energy of the light-cured ceramic slurry (influenced by the nature of the ceramic itself)

$S_d$  -- photosensitive parameters (affecting the nature of ceramic particles, optical properties of light-curing resin materials)

$C_d$  --- the layer thickness that can be formed by light curing at this exposure energy.

### 4.3. Degreasing and sintering of alumina ceramic light-curing slurry

(1) Degreasing. Remember not to degrease directly in the air, which will accelerate the rate of organic pyrolysis, increasing the incidence of porosity and other defects. So in order to reduce the occurrence of porosity must be degreased in a vacuum to reduce the rate of organic pyrolysis, so that the organic pyrolysis of the carbon generated in the vacuum fully pyrolysis and finally transformed into gas escape, so as to completely remove and reduce the purpose of porosity, cracks and other defects. The degreasing process flow is as follows: pyrolysis in a vacuum environment and expel gas (gas produced by organics) → input the right amount of air to oxidize with residual carbon (oxygen in air reacts with carbon elements) → remove impurities [5].

(2) Sintering. First, the temperature is ramped up to 1400°C at a rate of 10°C/min, held for 120 min then ramped up to 1600°C at a rate of 5°C/min, continued to be held for 120 min, and finally slowly ramped up to 1700°C at a rate of 2°C/min, held for 300 min to make the ceramic body fully densified [6].

## 5. Alumina ceramic light-curing slurry influencing factors and optimization methods

### 5.1. Influencing factors

Including dispersant type, dispersant concentration, solid content, and degreasing method, the specific influences are as follows.

(1) The type of dispersant directly affects the dispersibility and stability of the slurry. For example, dispersants such as sebacic acid, silane coupling agent KH550 and polymer KOS110 all affect the dispersibility and stability of alumina ceramic light-curing slurry to different degrees.

(2) The dispersant concentration and the solid content of the slurry directly affect the viscosity of the slurry. Many studies have confirmed that the polymer KOS110 as dispersant applied to alumina ceramic light-curing paste preparation and sintering will have different degrees of viscosity decline, KOS110 dispersant is too low will reduce the adsorption of alumina powder, thus failing to improve the viscosity of the role; dispersant concentration is too high will cause the powder to appear a large number of free, increasing the internal friction of the slurry, so that the slurry viscosity Excessive increase, affecting the curing process. The solid content of the slurry is also directly slurry viscosity. For example, the premise of ensuring the solid content of the slurry to properly reduce the viscosity, not only can effectively improve the dispersion of the slurry, but also enable the slurry to quickly adapt to the photoassimilation printing requirements, and improve and printing accuracy [7].

(3) Degreasing method directly affects the sintering of the slurry. For example, direct sintering in air will accelerate the rate of organic pyrolysis, and although the slurry can be fully reacted, it will also generate a large amount of gas and can not escape in time, increasing the incidence of defects such as holes. In contrast, degreasing sintering in vacuum can form internal and external pressure, so that the gas generated in the reaction process can be discharged in time to reduce the occurrence of defects such as holes.

### 5.2. Optimization method

(1) Optimization method of slurry dispersion and stability. First, according to the relevant requirements and standards to change the surface potential of ceramic particles, and reduce the mutual repulsion and friction between particles through electrostatic effect, for example, in the use of dispersant sebacic acid. For example, when using dispersant sebacic acid, sebacic acid and other dispersants to modify the surface of the powder, there will be part of the carboxyl group and the powder surface ion chemical reaction, so that the atoms form ionic bonds, thereby reducing the surface energy of the powder and the viscosity of the slurry, it is necessary to reduce the mutual repulsion and friction between the particles through electrostatic effect, so as to reduce contact collisions and improve the stability of the slurry. Secondly, by using silane coupling agent KH550 to

increase the thickness of the adsorption layer on the surface of the particles, thus increasing the spatial site resistance, finally anchoring the particles, promoting the dispersion of the powder in the slurry, making the surface of alumina organic, reducing particle collisions, and improving the dispersion and stability of the slurry. Third, the electrostatic effect is combined with spatial site-resistance to form a stabilization mechanism, such as using dispersants such as sebacic acid, silane coupling agent KH550 and polymer KOS110 to improve slurry dispersion and stability [8].

(2) Optimization methods of slurry viscosity. First, select the dispersant with appropriate concentration according to the technical standards and related requirements for the preparation of alumina ceramic light-curing slurry. Taking polymer KOS110 dispersant as an example, KOS110 dispersant at different concentrations has different modification effects on alumina particles; as the concentration of KOS110 dispersant increases, the adsorption of alumina particles on the surface of the dispersant will increase with it, and it is also easy to form an envelope layer and form a mesh structure in the resin, effectively avoiding mutual collision between particles and reducing the friction within the slurry and reducing the system viscosity; when the concentration increases to 7wt%, the slurry viscosity curve will rebound, at which time the adsorption of alumina particles to dispersant reaches saturation, thus destroying the stable structure between the powder and resin, triggering flocculation and leading to an increase in viscosity. Therefore, KOS110 dispersant with a concentration of 5 wt% is chosen as much as possible, and the dispersant at this concentration has the best effect on the modification of alumina particles, which not only improves the viscosity of the slurry, but also improves the dispersibility and stability of the slurry [9]. Secondly, the solid content of the slurry is positively correlated with the slurry viscosity and printing accuracy, i.e., the higher the solid content of the slurry, the higher the viscosity and printing accuracy, and to ensure the densities and properties of the ceramic body in the later degreasing and sintering process, the solid content of the slurry must be increased appropriately according to the actual, and the best alumina ceramic slurry solid content is 45 vol% under certain conditions of shear rate.

(3) Sintering optimization method. Including air sintering and vacuum sintering two methods, direct sintering in the air gas can not escape in time, so it is easy to appear holes and other defects; degreasing sintering in a vacuum can be timely gas discharge, but there is also a lack of oxygen, residual carbon is easy to react with the ceramic to generate miscellaneous and so on, so whether air sintering or vacuum sintering need to be optimized before operation, for example, on the basis of vacuum degreasing For example, two-step degreasing can be performed on the basis of vacuum degreasing (the principle is that after the first step of degreasing, the billet is placed in an air environment and warmed up to 600°C for the second step of degreasing), which is more effective in removing residual carbon than air degreasing, and the sintering quality and density are higher, which can effectively improve the ceramic properties [10].

## 6. Conclusions

Light-curing slurry preparation and degreasing of alumina ceramics is the main link in the production of alumina ceramics, which is directly related to the quality and quality of alumina ceramics, i.e., the higher the process level of this link, the higher the quality and quality of alumina ceramics, and vice versa, so the process level of light-curing slurry preparation and degreasing of alumina ceramics must be continuously improved. The previous alumina ceramic light-curing paste preparation and degreasing process still has many shortcomings, such as material selection is not up to standard, degreasing process professional level to be improved, etc., resulting in ceramic surface porosity, cracks and other defects, serious cases will directly damage the internal structure of the entire ceramic, thus difficult to shape, and ultimately become a failure of ceramic works. Therefore, the above analyzes the preparation and degreasing of alumina ceramic light-curing paste based on the understanding of alumina ceramics, and discusses the influencing factors and optimization methods of alumina ceramic light-curing paste.

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