Study on the Production Process of 304L Stainless Steel Injection Molding

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Abstract. 304L stainless steel powder injection molding was used to product the fiber connectors in this paper. The effects of different reaction parameters on 304L stainless steel powder injection molding, debinding process and catalytic effect of the sintering process were researched. The results show that: the injection parts had no obvious cracks, burrs and defects such as holes, good shape, smooth surface under 304L stainless steel powder system in the temperature of 185℃, molding temperature of 125℃, the injection rate of 90%, injection molding conditions of the injection pressure of 130MPa. The catalytic debinding process of injection molding, catalytic 4h at 120℃, nitrate gas into nitrogen gas rate was 2 L·h\(^{-1}\), the rate of 2.3 L·h\(^{-1}\), the debinding rate of debound parts was moderate, shape preserving was good. The qualified fiber connector joints could be obtained after the debound parts were sintered in vacuum, the sintering temperature was 1350℃, keeping 2 h.

1. Introduction

Metal powder injection molding (MIM) was a new technology which combines traditional powder metallurgy technology with plastic injection molding technology, and breaks through the limitation of traditional metal powder molding technology in product shape. At the same time, plastic injection molding technology can be used to produce parts with complex shapes in large quantities and high efficiency [1-3]. The metal powder and binder were mixed uniformly to form a rheological feed, and the part was formed by injection molding on the injection machine. The obtained part was densified by debinding and sintered into finished product. MIM, which can be directly formed into parts with complicated geometric shape. Usually 0.1g ~200g; good surface finish, high precision, typical tolerance of ±0.05 mm, good flexibility of alloying, wide range of application of materials, density of the product up to 95% ~99%, uniform internal organization, no internal stress and segregation; high degree of automation in production, no pollution, Cleaner production can be realized continuously in large quantities [4-6].

Powder injection molding 304L stainless steel has good candle resistance, widely used in mechanical, chemical, marine, automobile, instrument and other industries [7-8]. In order to ensure the advantages of the products, it was particularly important to control the quality of 304L stainless steel injection molding process. In this paper, the production process of the 304L stainless steel injection molded fiber connector joint was studied. The effects of different process parameters on the injection molding, catalytic debinding and sintering process of 304L stainless steel powder were studied.
2. Experimentation

The 304L stainless steel powder produced by Shijiazhuang Lide Powder material Co., Ltd., with an average particle size of 9 ~ 11 μm. The binder mainly consists of polyformaldehyde (POM), high density polyethylene (HDPE), polypropylene (PP) and a small amount of auxiliary additives. The formulation of binder was 85%POM+ 8%HDPE+ 7% (PP and other additives). The 304L stainless steel powder was mixed with binder according to a certain amount of powder, and then granulated on the granulator. The feed was added into the injection molding machine, and the optical fiber connector part was prepared. The drawing of the injection part was shown in figure 1.

![Figure 1. Schematic diagram of injection part](image)

The injection parts were placed in the catalytic debinding furnace and catalyzed by nitric acid to catalyze debinding. The debound parts was sintered in vacuum sintering furnace, which was divided into three stages: negative pressure debinding, vacuum internal sintering and forced cooling, and the 304L fiber connector joint was obtained.

3. Experimental results and analysis

3.1. Effect of injection molding process parameters on injection parts

The injection molding process was one of the key processes of MIM. The quality of the injection part directly affects the dimensional accuracy and mechanical properties of the final product of MIM [9]. In this process, defects such as internal pores, cracks and two-phase separation were easily produced, which can not be eliminated in subsequent processes. Therefore, controlling the injection molding process can improve the product yield and reduce the production cost. There were many factors affecting the injection molding process, the most important was the influence of injection parameters, including mold temperature, material temperature, injection pressure, injection rate and so on [10-11].

According to the density and appearance of the injection part, the injection parameters were adjusted and the ideal injection part was obtained. The state of the part under different injection parameters was shown in Table 2.

| Injection Temperature (°C) | Mold Temperature (°C) | Injection Rate (%) | Injection Pressure (bar) |
|---------------------------|-----------------------|--------------------|-------------------------|
| 170 ~ 180                 | 110 ~ 120             | 75 ~ 85            | 115 ~ 125               |
| Too low                  | Too high              | Too low            | Too high                |
| Too high                 | Too low               | Too high            |

It can be seen from Table 1 that too low injection temperature (material temperature 170 ~ 180 °C, mold temperature 110 °C ~ 120 °C) can lead to too high viscosity of the feed, which will lead to problems such as under injection, rough surface, low strength, etc. When the material temperature was 190 °C, the mold temperature was 130 °C. Too high injection temperature will lead to too low viscosity of the feed and larger shrinkage of the injection part during cooling will lead to the surface and internal pores. Too low injection rate (75% ~ 85%) will make the feeding filling cavity longer, resulting in a slight under injection of the part, and too high injection rate will make the gas in the mold cavity unable to be discharged in time, leading to the formation of pores in the injection parts. Burrs or even surface collapses and other defects. Too low injection pressure (115 bar ~ 125 bar) will lead to under injection and low relative density of injection part. Too high injection pressure (135 bar) will increase the friction between the feed and die, and lead to the difficulty of mold release. Under the
conditions of material temperature 185 °C, mould temperature 125 °C, injection rate 90 and injection pressure 130 bar, the injection parts has high relative density, good appearance and strength.

Table 1. Effects of injection parameters on the injection parts

| Material temperature °C | Mould temperature °C | Injection rate % | Injection pressure bar | Injection parts                                      |
|--------------------------|----------------------|------------------|------------------------|-----------------------------------------------------|
| 170                      | 110                  | 75               | 115                    | Low relative density, rough surface, low strength, fragile |
| 175                      | 115                  | 80               | 120                    | The relative density was low, the part was under injected, the part was not full, and the part was easy to break. |
| 180                      | 120                  | 85               | 125                    | Low relative density and rough surface               |
| 185                      | 125                  | 90               | 130                    | High relative density, no defect in appearance and smooth surface |
| 190                      | 130                  | 95               | 135                    | The relative density was high, there were holes, burrs and other defects on the surface of the part. |

3.2. Effect of catalytic debinding process parameters on debound parts

The binder must be removed before sintering. The purpose of debinding was to remove the binder in the shortest time without producing defects. This was a key step to maintain product shape and prevent defects such as product cracking. Catalytic debinding combines the advantages of thermal debinding and solvent debinding, and overcomes the disadvantages of traditional debinding, such as long time, many defects, high energy consumption and environmental pollution. By using POM, which was sensitive to acidic atmosphere and decomposed into formaldehyde in acidic atmosphere, the chemical stability of [13-14] HDPE can ensure that the debinding part has sufficient strength and does not deform. In order to improve the processability of the adhesive, it was necessary to add auxiliary additives such as plasticizer, such as PP, which were then evaporated or decomposed at a higher temperature [15]. Early studies have shown that the catalytic debinding rate can be controlled by controlling catalytic gas flow, debinding temperature and debinding time [16-18]. In this experiment, the debinding rate k was characterized by the following formula [19]:

$$k = \frac{(m_0 - m)}{m_0} \times 100$$  \hspace{1cm} (1)

Through comparative experiments, the debinding rate of debinding part under different catalytic debinding conditions was shown in Table 2. It can be seen from the table that with the increase of the catalytic debinding time, the debinding temperature and the flow rate of N₂, the debinding rate of the product increased. With the increase of the debinding temperature, the amount of debinding of the injection part increases, the temperature was below 110 °C, and the amount of debinding was small, but when the temperature was above 110 °C, the debinding speed of binder was faster, but it was found in the experiment that too high debinding temperature will result in deforming of debinding part, because too high temperature would lead to polymer softening, and too fast removal speed of binder would lead to defects. With the increase of catalytic gas flow rate, the concentration of gas in catalytic debinding furnace increased, thus accelerating the depolymerization of POM. Too low flow rate of nitric acid and low debinding rate affect subsequent sintering. If the concentration of nitric acid was too large, it will be corrosive to powder and equipment.
Table 2. Effect of catalytic parameters on the rate of debinding

| Debinding time (h) | Debinding temperature (℃) | Rate of HNO₃ (L·h⁻¹) | Debinding rate (%) |
|-------------------|---------------------------|-----------------------|-------------------|
| 1                 | 90                        | 1.0                   | 6.423             |
| 2                 | 100                       | 1.5                   | 6.740             |
| 3                 | 110                       | 2.0                   | 7.196             |
| 4                 | 120                       | 2.5                   | 7.305             |
| 5                 | 130                       | 3.0                   | 7.325             |

According to the requirements of powder loading and qualified product density, the debinding rate after debinding was the best in 7.3%, so the debinding parameters were set to 4 hours of debinding time, the debinding temperature 120℃, the HNO₃ entry rate was 2.5 L·h⁻¹, and the debound products have not found cracks, bubbles and other defects, and the SEM photos of the injection and skimmed parts under this parameter were shown in Figure 2. It was seen from the drawing that the particles were filled with the binder before the debinding, and the surface of the particle was covered with a flocculent binder. After the catalytic debinding, the floc disappears and the particle morphology was clearer. The body forms connected pores and was connected to the outside. Most of the binder has been catalyzed, and a few of the remaining binders will be in the skeleton. All the following sintering stages were removed.

![SEM of injection part and debound part](image)

(a) injection part  (b) debound part

Figure 2. SEM of injection part and debound part

3.3. Effect of sintering process parameters on sintered part

Sintering is the last step of MIM process. The removal of binder in debinding process leads to a large number of pores in debinding part. Although debinding part has certain sintering performance, in order to make the densification of debinding part as high as possible, higher comprehensive properties must be sintered at high temperature [20]. The catalytic debinding part was transported to the vacuum sintering furnace in vacuum and argon atmosphere. Under different sintering processes, the sintered bulk density and linear shrinkage were shown in Table 3.
Table 3. Effect of sintering process on sintered parts

| Maximum sintering temperature (℃) | Holding time (h) | Density (g·cm⁻³) | Linear shrinkage rate (%) |
|----------------------------------|------------------|------------------|--------------------------|
| 1330                             | 1                | 7.6252           | 13.85                    |
| 1330                             | 2                | 7.6543           | 13.92                    |
| 1350                             | 1                | 7.7985           | 14.20                    |
| 1350                             | 2                | 7.8074           | 14.23                    |
| 1370                             | 1                | 7.8217           | 14.35                    |
| 1370                             | 2                | 7.8279           | 14.36                    |

It can be seen from table 3 that the density of the sintered part increases with the increase of sintering temperature. It shows that increasing the sintering temperature was an effective measure to obtain higher density of the product. Compared with the density of the sintered part at 1350 ℃, the density of the sintered part at 1370 ℃ was less, indicating that the density of the sintered part in the continuous rising temperature was not changed, and the sintering temperature was too high. The grain coarsening and sinter shrinkage were not uniform, resulting in the deformation of the product, the reduction of the product performance and the increase of the energy consumption and the production cost. The linear shrinkage rate of the length direction of the sintered part increases with the increase of the sintering temperature. From table 3, it can be seen that the trend and density of the line shrinkage with the temperature were in accordance with the trend of the change of the sintering temperature.

Figure 3 shows the SEM of the sintered part fracture surface at the highest sintering temperature of 1350 ℃ for 2 hours. From the fracture surface SEM, it can be seen that the grain distribution of the sintered part was uniform and compact. It can be seen from the metallographic photographs that the microstructure was uniformly distributed, the grain boundary was smooth, the grain size was not abnormal, there were a few residual pores, but the size was small and the distribution was uniform, so it will not be the source of the crack. According to the application requirements of the product: the density of the sintered part was over 7.8 g·cm⁻³, the linear shrinkage was about 14%, and the appearance was not defective, so the best sintering temperature was 1350 ℃ and the heat preservation was 2 hours.
4. Conclusion

(1) Under the conditions of material temperature was 185 °C, mould temperature was 125 °C, injection rate was 90% and injection pressure was 130 bar, the injection part had a higher relative density, a better shape and a flat surface under the conditions of material temperature was 185 °C, mould temperature was 125 °C, injection pressure was 130 bar.

(2) The injection part was catalyzed at 120 °C for 4 h, the rate of nitric acid gas entry was 2 L.h⁻¹, and the nitrogen gas penetration rate was 2.3 L.h⁻¹. The debinding rate of the part was moderate and the shape preservation was good.

(3) Vacuum atmosphere sintering of debinding part, sintering temperature was 1350 °C, holding time was 2h, the qualified stainless steel fiber connector joint can be obtained.

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