Effect of mixing proportion and mixing time on primary slurry retention and surface roughness of investment casting shells

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Abstract : Surface quality of investment cast parts are mainly depends upon primary slurry condition which is used to make face coat. Zircon flour (ZrSiO$_4$) is widely used to make primary slurry. In this work different composition (75% - 25%, 70% - 30% and 65% - 35%) of zircon flour and colloidal silica is analyzed at different mixing time (at 24hrs, 48hrs and 72hrs). Viscosity, wettability, slurry retention, and surface roughness are measured and analysed. From the experimental work conclusion derived that composition with 75% zircon and 25% colloidal silica at mixing time of 72 hour gives best result in surface finish.

1. INTRODUCTION

Investment casting process is used to make casting products for many centuries. It is known as near-net shape process due to higher surface finish and good dimensional accuracy even in complex shapes. It is used for making of complex shapes of expansive metals where post processing is not affordable [1]. Principle of investment casting has been found to be used for rudimental items like jewellery, arts and idols in 5000 BC. Archaeologists investigated that Mesopotamia was the location of a civilised society who have skills in metallurgy and engineering, and also having knowledge to produce gold, silver and copper artefacts by investment casting. Investment casting process is widely used to make nozzles, turbine blades, vanes, valves and pumps and other items. In recent years, this process is found to be the most suitable to make body implants, due to its higher accuracy and surface finish [2, 3]. Due to poor machinability of Al metal matrix composite, investment casting is a potential option for making composite products [4].

Primary slurries to apply face coat are prepared by mixing flours with binder solution. Among the various primary coats applied with flours like SiO$_2$, Al$_2$O$_3$, and ZrSiO$_4$, shell made with zircon flour (ZrSiO$_4$) exhibits excellent surface morphology [5]. It was observed that with higher viscosity, thicker layer of primary coat was developed[6]. The ceramic retention (plate weight) of slurries increases with the increase in flour loading. The retention of slurry with higher flour to binder ratio provides thicker and smoother primary coat. Higher flour to binder ratio also improves the shell surface leading to better surface finish [7]. Higher viscosity binder with same ceramic flour to binder ratio provides higher viscosity of slurry, leading to higher suspensibility and retention. Optimal flour to binder ratio gives better surface finish [8].

Mixing proportion of flour with binder solution and mixing time while preparing primary slurry governs the slurry retention and surface finish of ceramic shell. In the present work zircon flour is...
mixed with colloidal silica binder by varying the mixing proportion and mixing time. The effect of mixing proportion and time is evaluated by measuring viscosity, slurry retention, wettability and surface roughness.

2. MATERIALS AND METHODS

2.1 Materials of slurries
The test slurries were prepared at room temperature (refer figure 1). Details of primary slurry (to apply face coat) composition and secondary slurry composition are shown in table 1 and table 2 respectively. Wetting agents are mixed in primary slurry to decrease its surface tension, generally only 0.5% agents are mixed with slurry [9]. The shells are made using conventional investment casting shell making procedure.

Table 1. Composition of primary slurry

| Composition no | Zircon flour - 350 mesh [ZrSiO$_4$] (%wt) | Colloidal silica (%wt) | Wetting agents (ml) |
|---------------|------------------------------------------|------------------------|--------------------|
| 1             | 75                                       | 25                     | 4                  |
| 2             | 70                                       | 30                     | 4                  |
| 3             | 65                                       | 35                     | 4                  |

Table 2. Composition of secondary slurry

| Ceramic powder (%wt) | Binder (%wt) | Wetting agent (ml) |
|----------------------|--------------|--------------------|
| Molochite powder 200 mesh [Al$_2$O$_3$·SiO$_2$] (45%) | Colloidal silica (55%) | - |

Figure 1. Slurry preparation.

2.2 Mould Preparation
Waxpatterns of dimension 7.5x7.5x1.5 cm$^3$ were prepared by developing rubber mould (refer figure 2). Wax in the form of granules was melt at 200ºC and poured inside the cavity of silicon mould and allowed to solidify. Total 36 patterns were prepared, 18 for retention test and 18 for making of mould. Viscosity, slurry retention rate, primary layer thickness and surface roughness were measured at 24 hours, 48 hours and 72 hours of mixing time.
The wax patterns were dipped in primary slurry and stuccoed with zircon flour of 110 mesh and allowed to dry for 6 hours to prepare face coat (refer figure 3). After drying of face coat, the moulds were dipped in secondary slurry and stuccoed with secondary sand of coarser mesh size and allowed to dry for 6 hours. This process was repeated for 6 times to prepare the 6 layers, and then these moulds were dipped in secondary slurry for seal coat. Stucco sizes used for preparing secondary layers are presented in table 3.

| Layer no | Stucco size in mesh |
|----------|---------------------|
| 1        | 50-80               |
| 2        | 30-80               |
| 3        | 30-80               |
| 4        | 16-30               |
| 5        | 16-30               |
| 6        | 16-30               |
| 7        | Sealing coat        |

These shells were allowed to dry for 12 hours before de-waxing. De-waxing process was done at 250°C temperature and then moulds were fired at 750 °C temperatures in resistance furnace (refer figure 4).

3. EVALUATION OF PRIMARY SLURRY

3.1 Viscosity Measurement
Viscosity of slurry was measured with Zahn B4 type cup (refer figure 5). Initially, cup was fully filled with slurry and orifice of cup was covered to stop the flow while filling the cup. Later, slurry was allowed to flow through the orifice and time taken to emptying the cup was recorded with stop-watch.
This test gives the time required to emptying the zahn B4 cup in seconds and it can be converted directly in different units of viscosity [10].

![Zahn B4 cup for viscosity measurement](image)

**Figure 5.** Zahn B4 cup for viscosity measurement

Viscosity of primary slurry is measured for each combination of mixing proportion and time. Five readings for each case are measured and average is considered and presented in table 4.

| Slurry mixing time | Composition | Average time of 5 test with Zahn B4 cup (sec) | Viscosity (Centipoise) |
|--------------------|-------------|-----------------------------------------------|------------------------|
| 24 hours 1 (75-25) | 23.6        | 295                                           |
| 2 (70-30)         | 17.3        | 206                                           |
| 3 (65-35)         | 12.5        | 135                                           |
| 48 hours 1 (75-25) | 28.2        | 360                                           |
| 2 (70-30)         | 19.2        | 232                                           |
| 3 (65-35)         | 14.6        | 168                                           |
| 72 hours 1 (75-25) | 33          | 440                                           |
| 2 (70-30)         | 22.3        | 283                                           |
| 3 (65-35)         | 14.8        | 172                                           |

It has been clearly seen that as the time of mixing increase viscosity also increases, this is due to the evaporation of water particles that are present in colloidal silica. The measurement also shows that as the amount of colloidal silica binder increases in, viscosity of slurry decreases subsequently.

### 3.2 Wettability Test

Wettability refers to the ease with which a liquid spreads across a solid surface. Contact angle (CA) is the main parameter introduced by Young that correlates wetting with interfacial tensions of the solid, liquid and vapour interfaces using simple considerations of the equilibrium at the triple line, i.e., the line where the solid, liquid and vapour phases come in contact as shown in figure 6 [11].

\[
\cos \theta = \frac{\gamma_{SG} - \gamma_{SL}}{\gamma_{LG}}
\]

Where, $\gamma_{SG}$ is solid-gas surface energy

$\gamma_{SL}$ is solid-liquid surface energy

$\gamma_{LG}$ is liquid-gas surface energy

$\theta$ is contact angle
Wettability of the slurry on the surface of wax pattern is measured with automated goniometer as shown in figure 7. A drop of primary slurry was placed on wax pattern which is placed on a testing platform. The shape of droplet is recorded with CCD camera and analyzed in the software to calculate contact angle as shown in figure 8.

Results of these test is presented in the table 5. It can be seen that as mixing time of slurry increased the contact angle increased. Increase in viscosity with slurry mixing time may responsible for these results. It was also observed from the measurements that the increase in amount of colloidal silica binder resulted in decreased viscosity which provides more spreading of slurry and contact angle get reduced.

| Slurry mixing time | Composition | Mean contactangle (in degree) |
|-------------------|-------------|------------------------------|
| 24 hours          | 1 (75-25)   | 28.3                         |
|                   | 2 (70-30)   | 22.7                         |
|                   | 3 (65-35)   | 17.3                         |
| 48 hours          | 1 (75-25)   | 32.1                         |
|                   | 2 (70-30)   | 26.1                         |
|                   | 3 (65-35)   | 18.6                         |
| 72 hours          | 1 (75-25)   | 32.3                         |
3.3 Slurry Retention Test

Slurry retention test is used to determine the amount of slurry that wax pattern can retain. In this test the weight of plate was measured before applying the face coat on wax pattern using digital weighing balance. Then weight of pattern was again measured after coating and drying. The amount of slurry that retained by wax pattern is calculated using following equation [7].

\[ R = \frac{(W_d - W_p)}{S} \]

Where,

- \( R \) = Retention rate in mg/cm²
- \( W_d \) = Plate weight before face coat in mg
- \( W_p \) = Plate weight after dipping and drying mg
- \( S \) = Surface area of plate in cm²

The thickness of primary layer \( H \) can be calculated form following equation [7].

\[ H = \frac{(W_d - W_p)}{DS} = \frac{R}{D} \]

Where, \( D \) = Density of Slurry.

Slurry retention test was performed on wax samples at all three conditions for all composition and compared. From this test it was found that in primary slurry higher amount of zircon gives higher retention, highest retention was observed in composition 1 and decreased in composition 2 and 3 (refer table 6). Retention also slightly increased with increase in mixing time. At 72 hours retention for composition 1 is 2.81 times higher than composition 3.

### Table 6. Slurry retention rate

| Slurry mixing time (In hrs) | Slurry retention Composition | Slurry retention rate (In mg/sq. mm) |
|----------------------------|-----------------------------|------------------------------------|
| 24                         | 1 (75-25)                   | 0.157                              |
|                            | 2 (70-30)                   | 0.105                              |
|                            | 3 (65-35)                   | 0.053                              |
| 48                         | 1 (75-25)                   | 0.161                              |
|                            | 2 (70-30)                   | 0.106                              |
|                            | 3 (65-35)                   | 0.055                              |
| 72                         | 1 (75-25)                   | 0.163                              |
|                            | 2 (70-30)                   | 0.107                              |
|                            | 3 (65-35)                   | 0.058                              |

Primary layer thickness was also measured with slurry retention rate, the results of thickness is presented in table 7. Primary layer thickness is increased with increased in mixing time; this is due to increase of viscosity which creates a dense thick layer on the pattern. Also as the amount of zircon is increased in primary slurry composition, thickness is increased due to increase in viscosity.

### Table 7. Primary layer thickness (mm)

| Composition | Mixing time (hrs) | 24 | 48 | 72 |
|-------------|-------------------|----|----|----|
| 1 (75-25)   |                   | 0.067 | 0.068 | 0.069 |
| 2 (70-30)   |                   | 0.048 | 0.049 | 0.052 |
| 3 (65-35)   |                   | 0.027 | 0.029 | 0.030 |
3.4 Surface Roughness measurement
Surface finish of moulds were measured with surface texture machine (refer figure 9) after firing of moulds and cutting a plate section from it as shown in figure 10. For measurement sample length 0.8 mm, pitch 0.5 μm and speed 0.1 mm/s was considered. Table 8 shows the average value of five measurements of surface roughness measured in micron for each combination of mixing proportion and time.

![Surface texture machine](image)

![Surface roughness measurement](image)

**Table 8. Surface Roughness (μm)**

| Composition | Mixing time (hrs) |
|-------------|------------------|
|             | 24   | 48   | 72   |
| 1 (75-25)   | 2.44 | 1.61 | 1.40 |
| 2 (70-30)   | 3.50 | 3.37 | 1.74 |
| 3 (65-35)   | 4.49 | 4.44 | 3.31 |

![Effect of mixing time on surface roughness for various compositions](image)

Comparison of surface roughness for each combination of mixing proportion and time can be observed from graph presented in figure 11. Surface roughness is decreased with increase in mixing time as more viscous slurry provides dense structure in primary coat. It was observed that composition 1 (75% zircon flour and 25% colloidal silica) gives best surface quality at mixing time of
72 hours with 1.4 micron Ra value.

4. CONCLUSION

From this work the following results can be concluded.

1. As the mixing time of slurry increased the viscosity of slurry is found to be increased. By increasing mixing time up to 3 times the slurry becomes more viscous by 50%, 37% and 27% for compositions 75-25, 70-30 and 65-35 respectively. This is due to evaporation of water particles from slurry which made it dense and viscous.

2. If the amount of zircon flour is increased in slurry composition by 10%, the viscosity of slurries get almost doubled.

3. It was observed that the increase in viscosity results in increased wettability, retention rate, primary layer thickness and surface finish.

4. From this work it can be seen that among all composition of primary slurry, composition 1 (75% zircon flour and 25% colloidal silica) at 72 hours of mixing time gives excellent results in retention rate and surface finish (improved up to 1.4 micron Ra value from 2.44 micron Ra).

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