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Effect of Slurry Composition on Plate Weight in Ceramic Shell Investment Casting

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This paper deals with the study of the effect of primary slurry parameters on the plate weight (ceramic retention test) in ceramic shell investment casting process. Four controllable factors of the zircon flour and fused-silica powder based slurries were studied at three levels each by Taguchi's parametric approach and single-response optimization of plate weight was conducted to identify the main factors controlling its stability. Variations in coating thickness with plate weight were calculated for each slurry and ceramic shell moulds were made on wax plate using primary slurry and coarse fused-silica sand as stucco. The Scanning Electronic Microscopy (SEM) technique has been used to study the surface morphology of zircon flour and fused silca powder particles as well as primary coating (shell surface). X-ray Diffraction (XRD) analysis was done to identify the various phases present in the ceramic slurry coating. Optical profilometer has been used to measure the surface roughness of the shells. The result reveals that the surface condition of shell can be improved by increasing the plate weight, corresponding to higher filler loading in the slurry. Confirmation experiments were conducted at an optimal condition showed that the surface quality of the ceramic shell mould were improved significantly. Castings were produced using Al-7%Si alloy in recommended parameters through ceramic shell investment casting process. Surface roughness of the produced casting were measured and presented in this paper.

Keywords coating thickness, plate weight, primary slurry, surface roughness

1. Introduction

Due to technological advances, ceramic shell investment casting process has sustained continued development and become the most versatile modern metal casting technique. In this process, the application of slurry coating and stucco (refractory particles) on wax pattern assembly is repeated, with drying between each successive coat, until a shell mould of sufficient thickness is achieved. The first layer is normally a fine coating so that a good surface finish on the casting will be obtained. Subsequent layers are made up of ceramic slurry and refractory granules. The shell will normally be made up of between five and eight layers depending on the cooling rate required and the subsequent metallurgical properties. Following completion of shell build, the expendable pattern material is removed usually by the action of heat and/or steam. The dewaxed mould is fired at a temperature of the order of 1000 °C, to develop the maximum ceramic bond and it is normal practice to cast the moulds immediately upon removal from the firing furnace and while they are still at an elevated temperature (Ref 1).

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The main features of investment casting ceramic shell mould quality are sufficient green (unfired) strength to withstand wax removal without failure and to withstand the weight of cast metal, respectively. The ceramic shell mould should be high thermal shock resistance to prevent cracking during metal pouring, chemical stability and have low reactivity with the metals being cast to improve the surface finish. The shell must have sufficient mould permeability, thermal conductivity, and low thermal expansion to maintain an adequate thermal transfer through the mould wall to allow the metal to cool. Finally, the shell must be easily collapsible after casting to facilitate the knocking out and the cleaning operation (Ref 2).

The quality of ceramic shells is dependent on the slurry and shell materials as well as the process by which the shells are built. Even a good slurry formula will not produce sound castings if the slurry is prepared in a substandard way. Poorly wet-in slurry will not develop its maximum strength potential and may result in serious shell problems such as shell cracking. Very few authors have discussed on the importance of the slurry formulation on the character of the ceramic granules and on the control procedures while noticing that it is a significant factor. Control procedures for slurries vary considerably among foundries, reflecting in part the wide range of specifications that different shops work to, depending on their product line. The most prevalent controls are the measurement of the initial ingredients and viscosity of the slurry.

The goal of any slurry makeup is to produce stable slurry. Stable slurry is a slurry that has achieved a given set of parameters such that it is in a usable condition that can be obtained time and time again on subsequent slurry makeup. In order for a slurry to be considered stable it must be well mixed to a point where the viscosity of the slurry is stable. Slurry mixing has a remarkable effect on the surface quality and

strength of the shell. There are many factors like silica type and level, refractory type, flour particle size distribution, solids loading, viscosity, plate weight, etc. that play a role on the final ceramic shell properties. Stability in handling of the cluster during coating and dewaxing, specific gas permeability and removal behavior are the demand of ceramic shell investment casting. Ceramic moulds with very low green strengths are prone to cracking during wax removal. The raw materials (refractory, binder, wetting agent, and antifoam) used to make slurry play a major role in determining the overall final ceramic shell characteristics.

Suitable choice of the ceramic materials can lead to smooth surface finish, high accuracy of the metal castings. Selection of any refractory filler material for shell making is dependent on a wide variety of factors, which can affect the properties of investment slurry, shell and casting and also the economy of the process (Ref 3). Various combinations of materials have been used to produce the ceramic mould, but due to its small particle size and chemical inertness with cast alloys, zircon is often used for the first coat while fused silica and alumino-silicates are used for the other shell coats. Roberts (Ref 4) showed that by using slurry of seven millimicron sol. containing fused-silica grains of three different particle sizes the resultant structure was stronger in both the green and fired states. Fused silica and alumina silica have become the two most widely used refractories for ceramic shell investment casting as a result of their relatively optimum characteristics of performance and material costs. Fused silica is particularly well suited as a shell refractory since its thermal expansion is less than half of that for alumina silica at 2,000 °C. Fused silica is lower in density, providing about 23% more volume per unit weight, which also makes lighter shells easier to remove after casting in the knockout and cleanup operations (Ref 5).

The refractory coating plays an intimate role in the ceramic shell investment casting process. It provides refractory protection to ensure no metal penetration and smooth surfaces of shell mould. It may also be used as a vehicle for the introduction of inoculants, designed to produce finer and more reproducible microstructures in the castings. Primary and secondary coat slurries have slightly different requirements for the manufacture of the mould. Secondary coats are required to build up the total shell thickness whereas primary coat slurries eventually provide the ceramic face of the mould in contact with the molten alloy. Primary Coating is one of the most important steps in the ceramic shell investment casting Process (Ref 6). The primary coat refractory must be nonreactive with binder used to produce slurry as well as with alloy being poured. Surface finish will be an important characteristic of the casting; great attention must be given to the nature of the ceramic filler. For this reason, slurry control takes on added significance in shell production.

The main factors determining the surface quality of the ceramic shell mould for investment casting of metal alloys include the density of the ceramic powder compact and viscosity of the primary coating slurry (Ref 7). The higher density fillers such as zircon or alumina will tend to sediment faster than alumino-silicate or silica fillers. Shrinkage of the gelled binders during drying tends to produce cracking of the coating. The complex physical phenomena occurring in the production of silicate bonded investment casting moulds, including shrinkage and cracking tendency in drying, have been examined in the detailed treatment by mills (Ref 8). Investments casting surface defects are primary coat related during the manufacture of an investment casting ceramic shell mould. Most water-based primary coats are composed of the colloidal silica binder and suitable ceramic filler. The slower drying of water based, as compared with alcohol based, silica binders are useful in that it allows sufficient time for the manipulating operation to ensure a smooth and even coating, and total surface coverage at the stuccoing stage (Ref 8). Temperature and humidity are critical factors in providing a consistent and even coating on the wax pattern. Slurry rheology is influenced by the shear characteristics of mixer used and refractory properties, i.e., density, morphology (particle shape), particle size, and distribution all control slurry rheology. The actual weight of slurry adhering to a standard test plate under controlled conditions is determined in ceramic retention test (plate weight) which is useful for controlling slurry coverage and its rheology or flow characteristics. Plate weight test for primary coats relates much more to their dipping and draining characteristics. The gain in weight represents the amount of slurry retained and hence the coatability of the slurry. It is mainly used on primary slurries but can also be used on backup slurries. The plate weight can influence the porosity or permeability of the primary coating and coating thickness, which can cause stucco penetration. Plate weight is one method used to indirectly measure thixotropy (Thixotropy defines how the slurry flows under changing shear conditions) and coating thickness. Plate weight test serves as a functional test to ensure the consistency of the slurry. Practically, as slurry drains and levels, it forms a film of defined thickness. A thixotropic slurry becomes more viscous as the shear decreases, leaving a thick film. The thickness of the primary slurry layer (coating) can be calculated from the equation (2) specified in Section 3.4.

2. Taguchi Design Method

Taguchi recommends a three-stage process: system design, parameter design, and tolerance design (Ref 9, 10). The Taguchi design method is a simple and robust technique for optimizing the process parameters. Design of experiment (DOE) has been a very useful tool to understand process characteristics and to investigate how inputs affect responses based on statistical backgrounds. In addition, it has been used to systematically determine the optimal process parameters with fewer testing trials. In this method, main parameters which are assumed to have influence on process results are located at different rows in a designed orthogonal array. With such an arrangement completely randomized experiments can be conducted. The optimal process parameters obtained from the Taguchi method are insensitive to the variation in environmental conditions and other noise factors. According to Taguchi's methodology, no matter how the quality of the product is measured, the quality characteristics are divided into three characteristics: target-the-best, larger-the-better, and smaller-the-better (Ref 11). In this method, signal to noise (S/N) is used to represent a response or quality characteristic and the largest S/N ratio is required. In the case of plate weight and viscosity, higher values of raw data are desirable. The S/N ratio for each level of process parameter is computed based on the S/N analysis. In general, we get a better signal when the noise is smaller; so that a large S/N ratio yields improved final results. Increasing the S/N ratio makes the final results more desirable. That mean the variance of the final results becomes smaller. Therefore, the optimal level of process parameter is the level of

highest S/N ratio. In the present work Taguchi's parameter design approach is used to study the effect of process parameters on the plate weight and primary coatings in ceramic shell investment casting process. From the S/N ratio, the effective parameters having influence on process results can be seen and the optimal sets of process parameters can be determined. Furthermore, a statistical analysis of variance (ANOVA) is performed to see which process parameter is statistically significant for plate weight property. The optimum condition for plate weight characteristic has been established through S/N data analysis aided by raw data analysis.

2.1 Selection of an Orthogonal Array (OA)

Before selecting a particular OA to be used as a matrix for conducting the experiments, the following two points must first be considered (Ref 9, 10):

1. The number of parameters and interactions of interest.
2. The number of levels for the parameters of interest.

The nonliner behavior, if exists, among the process parameters can only be studied if more than two levels of the parameters are used. Therefore, each parameter was analyzed at three levels. The selected number of process parameters and their levels are given in Table 1. For the sake of simplification, the second-order interactions among the parameters were not considered. Each three level parameter has 2 degree of freedom (DOF) (Number of levels—1), the total DOF required for four parameters each at three levels is 8 [=4×(3 − 1)]. As per Taguchi's method the total DOF of selected OA must be greater than or equal to the total DOF required for the experiment. So an L_9 OA (a standard 3-level OA) having 8 (=9 − 1) degree of freedom was selected for the present analysis (Table 2).

3. Experimental Study

3.1 Ceramic Materials

The choices of particular slurries will depend on the alloy and casting temperature being used. In the present investigations, primary slurries have been formulated using a mixture of zircon flour and fused-silica powder as filler and colloidal silica as binder. Mixture of triton (wetting agent) and *n*-octyl alcohol (antifoaming) used as Catalyst. All formulas are based on weight rather than volume and all components are weighed by electronic balance. The following properties have been studied to achieve well-stabilized ceramic slurries in this work.

3.2 Viscosity

Viscosity was calculated using the Brookfield DV-II + Viscometer (accuracy: ±1.0% of range, reproducibility: ±0.2%, speed: 0.01-200 rpm). All the values were measured at constant speed.

3.3 Density

Density of the slurry was determined with the use of conventional method by measuring mass and volume.

Table 1 Definition and trial levels factors in the Taguchi's L_9 OA experiment

Process parameter designation	Process parameters	Level 1	Level 2	Level 3
A	Zircon flour (g)	120	180	240
B	Fused silica (g)	60	120	180
C	Filler/sol ratio (g/mL)	2.5	3.0	3.5
D	Catalyst (mL)	2	3	4

Particle size 325 US mesh; Mixing time 25 h, Wax plate surface area 3-in square, sol: Colloidal silica 30%, Catalyst: 2 mL (50% *n*-octyl alcohol and 50% tri ton), Temperature: 30 ± 2 °C

Table 2 The L_9 (3^4) OA (parameters assigned) with response

		Parameters trial conditions				Plate weight (raw data) g/cm^2			
		A	B	C	D				
Sr. no.	Run order	1	2	3	4	R_1	R_2	R_3	S/N ratio (db)
1	3	1	1	1	1	0.123	0.122	0.124	−18.20
2	7	1	2	2	2	0.127	0.126	0.130	−17.88
3	5	1	3	3	3	0.126	0.130	0.131	−17.79
4	1	2	1	2	3	0.135	0.135	0.136	−17.37
5	4	2	2	3	1	0.133	0.134	0.135	−17.45
6	6	2	3	1	2	0.130	0.133	0.134	−17.56
7	9	3	1	3	2	0.145	0.144	0.146	−16.77
8	2	3	2	1	3	0.142	0.141	0.142	−16.97
9	8	3	3	2	1	0.143	0.142	0.144	−16.89

$\overline{T}$ = overall mean of plate weight = 0.134 g/cm^2
R_1, $R2$, $R3$ represent responses value for three repetitions of each trial. The 1s, 2s, and 3s represent levels 1, 2, 3, and 4 of the parameters, which appear at the top of the column. Y_{ij} are the measured values of the quality characteristic (response)

3.4 Plate Weight

The ceramic retention rate R was calculated using the equation

$$R = (\mathrm{Wd} - \mathrm{Wp})/S, \quad \text{(Eq 1)}$$

where Wp is the undipped plate weight, Wd the dipped weight of the plate, and S is the surface area of the plate (Ref 12). Plate weight was measured with electronic balance having a least count of 0.001 g. The thickness of the primary slurry layer H can be derived from the equation

$$H = (\mathrm{Wd} - \mathrm{Wp})/\mathrm{DS} = R/D, \quad \text{(Eq 2)}$$

where D is the density of the slurry.

3.5 Scanning Electronic Microscopy (SEM)

Scanning electronic microscopy used for high magnification study of metal surfaces. SEM analysis was done using the JEOL-T20 with 500 and 1000 magnifications. The samples were ultrasound deaglomerated in etalon for 10 min, and then gold spattered.

3.6 X-Ray Diffraction Analysis (XRD)

The XRD analysis was carried out with Bruker AXS D8 Advance Diffractometer (Germany) with copper anticathode radiation. Work pressure was 42 kV, power rate I = 30 mA, and the samples were tested in the range of 20-110 °C.

4. Slurry Mixer

The main objective of slurry mixer is to thoroughly mix the refractory filler and binder to produce stable viscosity of the slurry for a given set of parameters. The design and agitation characteristics of the mixing tank will ensure full dispersion in order to avoid sedimentation and entrapped air to maintain the homogeneity of the slurry.

Figure 1 shows the schematic moderate shear propeller type slurry mixer equipment. This arrangement is equipped with a speed controller that provides adequate shearing action to break up agglomerates and strips the air off particle surfaces allowing the slurry to stabilize in a few hours. The shear mixing has shortened the time required to completely wet in the slurry. The actual stabilization time is dependant upon the mixing speed and slurry composition. An additional feature is the use of a timer that turns the mixer on and off for a short period of time (typically 10 min on and 5 min off). This helps the air which is trapped in the slurry to migrate to the surface and reduces the heat buildup in the slurry due to friction.

4.1 Wax Plate Coating and Production of Casting

Details of slurry composition and their levels are given in Table 1. In order to achieve consistency of slurry characteristics correct preparation and maintenance must be ensured. The mixing method used in this study was the same, i.e., ingredients were placed in a steel tank and rotated. A nonionic wetting agent (triton) and *n*-octyl alcohol as an antifoam agent are used to ensure good coverage of the wax plate. The air-dried water based binder system is used for wax plate coating. All dipping was carried out under ambient conditions, by keeping dipping,

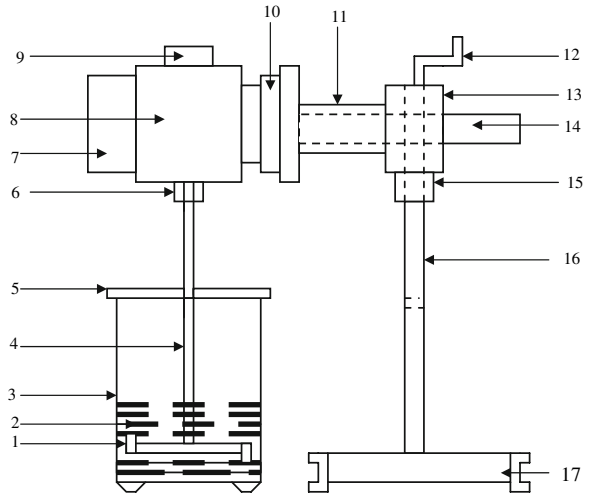

Fig. 1 Slurry mixer: (1) impeller eye, (2) slurry, (3) steel tank, (4) impeller rod, (5) cover plate, (6) fixture, (7) speed controller, (8) electric motor, (9) timer, (10) coupling plate, (11) horizontal barrel, (12) lever, (13) vertical barrel, (14) extension rod, (15) lock nut, (16) stand, and (17) base channel

draining and drying time 30 s, 60 s and 4 h, respectively. Drying of the liquid coating was carried out by evaporative means. Three shells (7-9 mm thick) are produced as usual practice from each slurry using calcined fused silica as back up stucco (mesh sizes −16 + 30 and −30 + 80). In the shell building process for casting, the coated wax pattern was stuccoed with zircon sand (AFS No. 120) by the rainfall sanding method. The pattern was rotated gradually to achieve an even coating of stucco material, which adheres to the surface of the wet slurry. This coat was dried at a temperature of 40 °C, 50% relative humidity for 4 h. After drying, secondary coat was applied by dipping primary coated pattern in fused slica backup slurry and coarse fused silica backup stucco was used. Each secondary coat was dried at a temperature of 40 °C, 50% relative humidity and for 2 h. This secondary coating and stuccoing process are repeated four times, to form a self-supporting shell.

For dewaxing, the shells was turned upside down and placed in a flash firing oven at a temperature 550 °C where the wax melts and pours out through the gate and pouring cup. The remaining ceramic shell molds are fired at 900 °C or 1 h to burn out the last traces of wax, to develop the high temperature bond of the ceramic system, and to preheat the mold in preparation for casting. The ceramic shell mould obtained after firing is shown in Fig. 2. The optical profilometer (Wyko NT1100) was used to measure surface roughness values of the internal surface of ceramic shell, i.e., the face coat surface after high temperature firing. Several Ra values were taken at different locations on the shell surface. Al-7%Si alloy (650 °C) was poured in the self-supported shell moulds and castings of the test pattern were produced as shown in Fig. 3. After the casting, all refractory material on the cast test piece was carefully removed by moderate-pressure water blast, and very light rubbing with wire brush/knife. On the basis of visual observations, the aluminum alloy castings were shiny and it can be said that there is a complete absence of mold-metal reactions in aluminum castings. Surface roughness of the produced castings is presented in Table 3.

5. Analysis and Discussion

The slurry plate weight was used as a response parameter in the present work. The values of plate weight for each trial condition and replications were recorded and are given in Table 2. The slurry formulation experiments were performed at the trial conditions shown in Table 2. Three samples of slurry for each trial condition was prepared using randomized technique and the run numbers have been given in the second column of the Table 2. The quality characteristic for slurry plate weight is of higher-the-better type. So, the S/N ratio for the "higher-the-better" type as given below was used:

$$\left(\frac{S}{N}\right)_{HB} = -10\log\left[\frac{1}{n}\sum_{j=1}^{R}(1/Y_j^2)\right], \qquad \text{(Eq 3)}$$

where Y, $j = 1, 2, \ldots n$ are the response values for the trial conditions repeated "n" times.

Fig. 2 Ceramic shell after firing

Fig. 3 Castings of Al-7%Si alloy

The S/N ratio was computed for plate weight for each of the nine trial values. These values are also given in Table 2. The viscosity and density values of slurry were also taken at different depths of the slurry test samples. Variations in coating thickness with plate weight was calculated for each slurry and shown in Table 3. The mean response refers to the average value of the performance characteristic for each parameter at different levels. The average values of plate weight for each parameter at levels 1, 2 and 3 were calculated and are given in Table 4. These values are plotted in Fig. 4. The main effects (raw data) of the various process parameters when they change from lower to higher levels are also given in Table 4 and are shown in the Fig. 4. The average values of S/N ratios of various process parameters at different levels are given in Table 5. These values are also plotted in Fig. 4. As shown in Fig. 4(a), the S/N ratio increases quickly as the zircon flour increases from 120 to 240 g and it is maximum when the zircon flour is 240 g. From the figure, it is clear that the plate weight achieved by higher quantity of zircon flour is significantly higher than that achieved by less quantity of zircon flour. The zircon flour has a positive effect on the plate weight of the slurry and, therefore, a larger value is desired, the optimum of which is found to be 240 g. Increase in plate weight with the increase of zircon flour has been attributed to the presence of more number of fine particles at level three of zircon flour. Higher solids loading in slurry will reduce the shrinkage during drying of coating and increase the green strength which will help to prevent shell cracking during dewaxing operation. As shown in Fig. 4(b), the S/N ratio increases slowly as the fused silica increases from 60 to 120 g and it is maximum when the fused silica is 180 g. This indicates that the amount of difference noted in plate weight is dependent on the flour loading, as the change in quantity of flour change the relative percentages of mixture. Zircon flour and fused-silica mixture slurries have shown good flowability on the wax pattern. For filler/sol ratio,

Table 3 Properties of slurry and coating thickness

Sr no.	Run order	Mean Density (gm/cm^3)	Mean viscosity (cp)	pH value	Mean plate weight (g/cm^2)	Coating (Primary) thickness (mm)	Mean surface roughness (μm)	
							Coating	Casting
1	3	1.69	1508	9.5	0.1230	0.73	2.05	2.95
2	7	1.78	1545	9.3	0.1276	0.72	1.97	3.16
3	5	1.86	1603.333	9.4	0.1290	0.70	1.73	2.71
4	1	1.82	1683	9.3	0.1353	0.74	1.58	3.24
5	4	1.86	1783.333	9.5	0.1340	0.72	1.87	3.24
6	6	1.92	1822.333	9.4	0.1323	0.68	1.64	3.02
7	9	1.93	1929	9.3	0.1450	0.75	1.03	2.03
8	2	1.97	1956.667	9.5	0.1416	0.72	1.51	2.30
9	8	2.03	2073	9.4	0.1430	0.70	1.24	2.19

Table 4 Main effects of plate weight on raw data

Process parameter	Level	Zircon flour (A)	Fused silica (B)	Filler/sol ratio (C)	Catalyst (D)
Average values	L1	0.1265	0.1344	0.1323	0.1333
	L2	0.1338	0.1344	0.1353	0.135
	L3	0.1432	0.1347	0.136	0.1353
Main effects	L2-L1	0.0073	0	0.003	0.0016
	L3-L2	0.0093	0.0003	0.0006	0.0003
Difference	{(L3-L2) − (L2-L1)}	0.002	0.0003	−0.0023	−0.0013

L1, L2, and L3 represent average values raw data at levels 1, 2, and 3, respectively, of parameters. L2-L1 and L3-L2 are the average main effect when the corresponding parameter changes from level 1 to level 2 and level 2 to level 3, respectively

Table 5 Main effects of plate weight on S/N data

Process parameter	Level	Zircon flour (A)	Fused silica (B)	Filler/sol ratio (C)	Catalyst (D)
Average values	L1	−17.9583	−17.4491	−17.582	−17.5181
	L2	−17.4664	−17.4379	−17.3822	−17.4075
	L3	−16.8805	−17.4181	−17.3411	−17.3795
Main effects	L2-L1	0.4919	0.0112	0.1998	0.1106
	L3-L2	0.5858	0.0198	0.0410	0.0279
Difference	{(L3-L2) − (L2-L1)}	0.0939	0.0086	−0.1588	−0.0827

L1, L2, and L3 represent average values raw data at levels 1, 2, and 3, respectively, of parameters. L2-L1 and L3-L2 are the average main effect when the corresponding parameter changes from level 1 to level 2 and level 2 to level 3, respectively

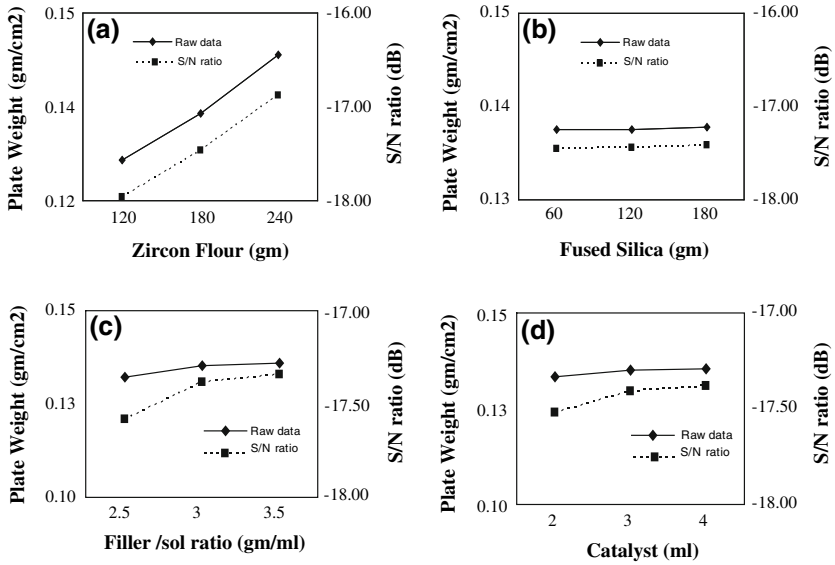

Fig. 4 Effects of process parameters of (a) zircon flour, (b) fused silica powder, (c) filler/sol ratio, (d) catalyst on plate weight (raw data), and S/N ratio (main effects)

the S/N ratio increases slowly as the filler/sol ratio increases from 2.5 to 3.5. This is due to the increase in filler loading in the slurry. Fig. 4(c) shows that a higher filler/sol ratio has a positive influence on the plate weight of the slurry. It has been found that slurry with filler to sol ratio higher than 3.5 g/mL could not be obtained in a stable form. The S/N ratio and plate weight of slurry is maximum at third level of parameter D_3 (catalyst), which means that 4 mL is the optimum value of the catalyst in the 2-4 mL range. Figure 4(d) shows that the S/N ratio increases as the catalyst level increases; this may be due the antifoaming action of catalyst to release entrapped air in the slurry, and partly due to the presence of triton in catalyst which increases the wetting properties of the slurries. This reveals that the catalyst affects plate weight of slurry positively and therefore, effectively improves the stability of slurries. It is clear from Fig. 4 that the slurry plate weight is highest at the third level of parameters A (A_3), third level of the parameter B (B_3), third level of the parameter C (C_3) and the third level of parameter D (D_3). The S/N ratio analysis (Fig. 4) also suggests the same levels of the parameters (A_3, B_3, C_3, and D_3) as the best levels for maximum plate weight of ceramic slurry.

In order to study the significance of the process parameters toward the plate weight, ANOVA was performed. The pooled versions of ANOVA of the raw data and the S/N data for plate

weight are given Tables 6 and 7, respectively. From these tables, it is clear that the parameters A, C, and D affect the raw data and S/N ratio significantly for plate weight values. The parameters and their selected optimal levels are found to be as:

Zircon flour (A_3) = 240 g.
Fused silica (B3) = 180 g.
Filler/sol ratio (C3) = 3.5
Catalyst (D3) = 4 mL.

Therefore it has been concluded that the ultimate coating requires optimal feed characteristics such as the initial solid content or the viscosity of the slurry and well-controlled process to optimize system benefits and attributes.

Figure 5(a) and (b) shows the surface morphology of the particles of zircon flour and fused silica, respectively, on the same magnifications 1000×. It is evident from Fig. 5(a) that zircon flour has rough surface with plate shape particles having average particle size 26.34 microns. Whereas, fused silica (Fig. 5(b)) consists of very fine particles of irregular shape with average particle size 5.73 microns. The particles of zircon flour are highly angular, and are larger than fused silica. Fused silica appeared to be more equiaxed, and large agglomerates can be observed. These agglomerates have been broken up and dispersed during mixing of slurries, producing stable systems. Zircon flour has a high density which facilitates allowing the shell material to settle into the finer details of a pattern and fine particles also prevent settling of the slurry when not in use.

The scanning electronic microscope micrograph showing the surface morphology of the shell (primary coating) made from slurry with optimum plate weight is shown in Fig. 6. This micrograph shows that the shell is having a uniform, smooth, and homogenous appearance with well-dispersed large and small particles. SEM micrograph of primary coating surface

Table 6 Pooled ANOVA—raw data

Source	SS	DOF	*V*	*F*-Ratio	SS′	*P* %
Zircon Flour	0.00126	2	0.000628	308.85*	0.001251481	90.25
Fused Silica	6.6667E–07	···	···	···	···	···
Filler/Sol ratio	6.8667E–05	2	3.43E-05	16.88*	6.41481E-05	4.62
Catalyst	2.0667E–05	2	1.03E-05	5.08*	1.61481E-05	1.17
E (Pooled)	4.0667E–05	20	2.03E-06		5.42222E-05	3.91
Total (*T*)	0.00138667	26	···	···	0.001386	100

*Significant at 95% confidence level
SS = Sum of Squares, DOF = degree of freedom, V = variance, SS′ = Pure Sum of Squares

Table 7 Pooled ANOVA—S/N data

Source	SS	DOF	*V*	*F*-Ratio	SS′	*P* %
Zircon Flour	1.7470746	2	0.873537	1176.86*	1.7456	92.82
Fused Silica	0.0014845	···	···	···	···	···
Filler/Sol ratio	0.0996567	2	0.049828	67.13*	0.0981	5.22
Catalyst	0.0322369	2	0.016118	21.71*	0.0307	1.63
E (Pooled)	0.0014845	2	0.000742	···	0.0059	0.31
Total (*T*)	1.8804528	8	···	···	1.8804	100

*Significant at 95% confidence level
SS = Sum of squares, DOF = degree of freedom, V = variance, SS′ = pure sum of squares

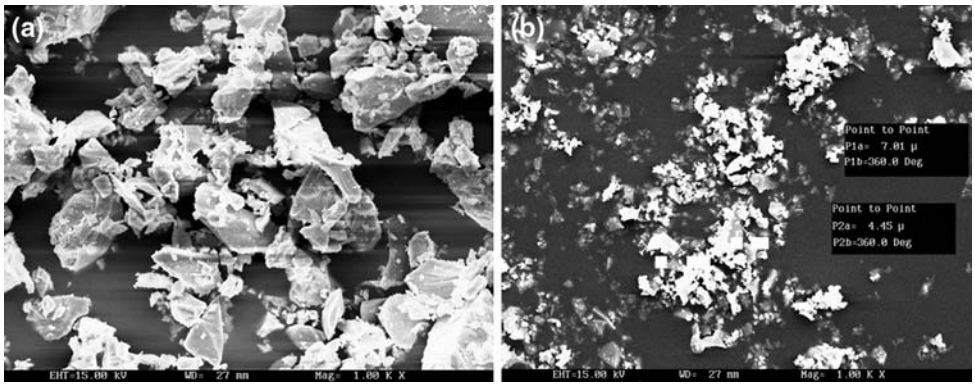

Fig. 5 (a) SEM of (a) zircon flour and (b) fused silica powder

shows (Fig. 6) that coating sample contains with homogeneously distributed fine pores that will help to remove gases formed during casting. Porosity can clearly be seen in SEM picture with ×1000 magnification. Dark black spots indicate the porosities in the coatings. From the SEM analysis, it is clear that particles of different grain size contribute better uniform and continuous coating on pattern; this may be due to the cohesion between the particles. Among the numerous parameters that influence the coating quality, the filler powder morphology has a great importance. The surface roughness of the shell (primary coating) was also measured by optical profilometer, which comes out to be 1.09 μm and ceramic shell mould surface obtained after firing is shown in Fig. 2. Therefore, the smoothness of the shell surface as indicated by SEM micrograph has been confirmed as the shell is having a very low surface roughness value of 1.09 μm. It can be concluded that higher plate weight slurry has useful in producing a superior shell surface with good homogeneity. In addition the coating is free from defects such as agglomerates and cracks. X-ray analyses have been performed on the prepared coating samples are shown in Fig. 7. Figure 7 shows that the coating sample has $ZrSiO_4$, TiO_2, Fe_2O_3, Al_2O_3, MgO, and SiO_2 phases. The most prominent phase of coating samples is $ZrSiO_4$ as indicated in Fig. 7. Further, it is found that there is no inter-metallic phase formed in the coating. XRD analysis indicated the absence of impurity elements such as silver, lead, and bismuth that can degrade cast metal properties.

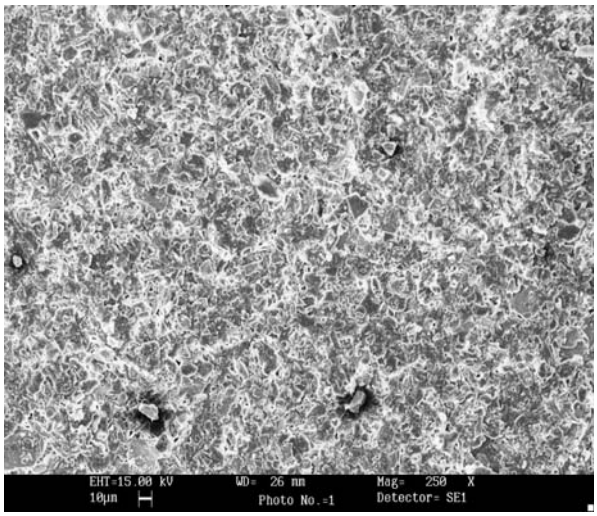

Fig. 6 SEM coating sample

6. Estimation of Optimum Performance Characteristics

The optimum value of plate weight is predicted at the selected levels of significant parameters A_3, C_3, and D_3 (Table 6). The estimated mean of the response characteristic plate weight (g/cm^2) can be determined (Ref 13) as

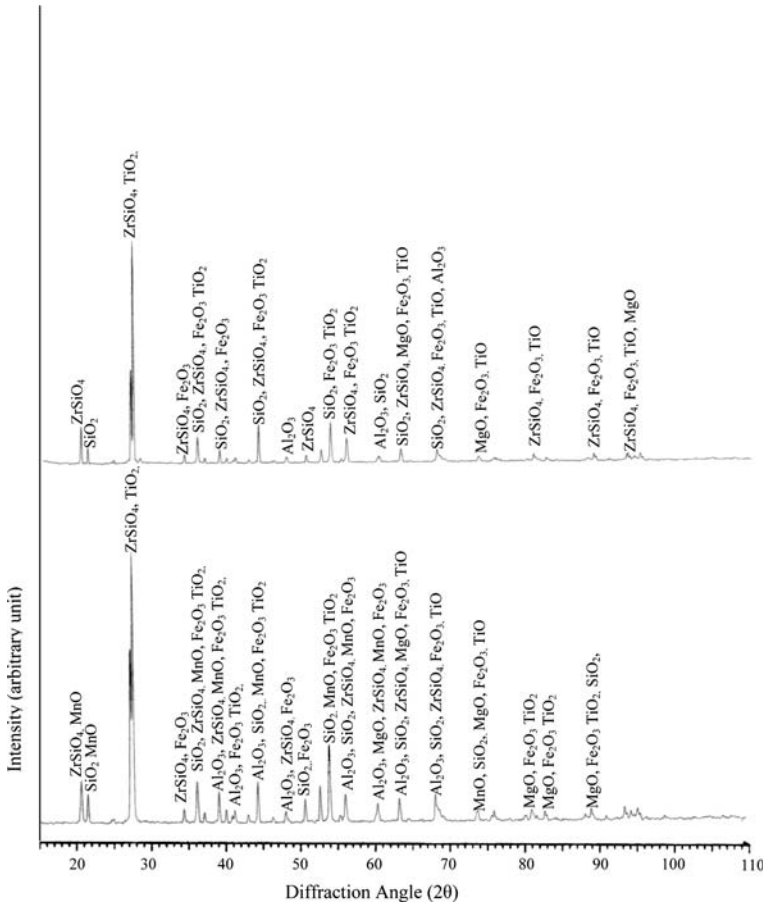

Fig. 7 X-ray diffraction for the two coating samples

$$\text{Plate weight} = \overline{A_3} + \overline{C_3} + \overline{D_3} - 2 \times \overline{T}, \quad \text{(Eq 4)}$$

where $\overline{T}$ [t1] = overall mean of plate, weight = 0.134 g/cm^2 (Table 2), $\overline{A_3}$ = average plate weight at the third level of zircon flour = 0.143, $\overline{C_3}$ = average plate weight at the third level of filler/sol ratio = 0.136, and $\overline{D_3}$ = average plate weight at the first level of catalyst = 0.135.

Substituting the values of various terms in Eq. (4),

$$\text{Plate weight} = 0.143 + 0.136 + 0.135 - 2 \times 0.134 = 0.146\,\text{g/cm}^2.$$

The 95% confidence interval of confirmation experiments (CI_{CE}) and of population (CI_{POP}) was calculated by using the following equations:

$$CI_{CE} = \sqrt{F_\alpha(1, f_e) V_e \left[\frac{1}{n_{eff}} + \frac{1}{R}\right]} \quad \text{(Eq 5)}$$

and

$$CI_{POP} = \sqrt{\frac{F_\alpha(1, f_e) V_e}{n_{eff}}}, \quad \text{(Eq 6)}$$

where $F_\alpha(1, f_e)$ = The F ratio at the confidence level of $(1-\alpha)$ against DOF 1 and error degree of freedom f_e.

$$n_{eff} = \frac{N}{1 + [\text{DOF associated in the estimate of mean responce}]} = 3.857$$

N = total number of results = 27 (treatment = 9, repetition = 3), R = sample size for confirmation experiments = 3, V_e = Error variance = 2.03E−06 (Table 6), f_e = error DOF = 20 (Table 6), and F0.05 (1, 20) = 3.4928 (tabulated F value).

So, CI_{CE} = ±0.00205 and CI_{POP} = ±0.00136.

The predicted optimal range (for a confirmation runs of three experiments) is:

$$\text{Mean plate weight} - CI_{CE} < \text{plate weight}(\text{g/cm}^2) < \text{mean plate weight} + CI_{CE}$$

$$0.14395 < \text{plate weight } (\text{g/cm}^2) < 0.14805$$

The 95% conformation interval of the predicted mean is:

$$\text{Mean plate weight} - CI_{POP} < \text{plate weight } (\text{g/cm}^2) < \text{mean plate weight} + CI_{POP}$$

$$0.14464 < \text{plate weight } (\text{g/cm}^2) < 0.14736$$

7. Confirmation Experiments

Three confirmation experiments were conducted at the optimum composition of the slurry parameters. The zircon flour was set at the third level (A_3), fused-silica powder at the third level (B_3), filler/sol ratio at third level (C_3), and catalyst was kept at the third level (D_3). The average plate weight was 0.1442 g/cm^2, which are within the confidence interval of the predicated optima of plate weight.

8. Conclusions

Ceramic slurry can be considered as one of the major component for making investment shell molds of precision components. The effect of slurry parameters on the plate weight was evaluated with help from the Taguchi method. The filler/sol ratio was a dominant parameter for superior coating and the next was the catalyst. Optimal slurry conditions to maximize the plate weight were determined within the range of test conditions employed; the following conclusions have been drawn:

1. The plate weight of the slurries gradually increased with increasing filler loading. The plate weight of the slurry with a filler/sol ratio (g/mL) of 3·5 was around 0.1442 g/cm^2 and provider of thicker and smoother primary coat.
2. The optimal levels of Zircon flour, fused silica, filler/sol ratio, and the catalyst for maximum plate weight correspond to the highest S/N ratio and Zircon flour, filler/sol ratio and the catalyst significantly affects the slurry plate weight.
3. The predicted optimal range for plate weight has been found to be as 0.14395 < Plate weight (g/cm^2) < 0.14805.
4. The 95% confidence interval of the predicted mean for plate weight is 0.14464 < Plate weight (g/cm^2) < 0.14736.
5. Major factors which contribute to slurry performance and stability are filler size and particle distribution.
6. The surface condition of primary coat, i.e., shell surface can be improved (surface roughness = 1.09 μm) by increasing the filler/sol ratio in the slurry to 3.5.
7. The recommended optimum parameters can be successfully used to produce Aluminum alloy investment castings. The surface smoothness varying within the range of 2.03-3.24 μm was obtained by using the ceramic shell investment casting process.

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