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ORIGINAL ARTICLE

Influence of Slurry Composition on Mould Properties and Shrinkage of Investment Casting

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Abstract Ceramic shell mould plays a key role in achieving dimensional accuracy and quality of casting. This work investigates the effect of mould parameters such as silica concentration, filler to binder ratio and the number of secondary coats on mould properties, namely modulus of rupture, adjusted fracture load, and permeability. Experimental results report that enrichment in silica results in drastic growth in adjusted fracture load with a small increase in modulus of rupture and no change in permeability. Improvement in filler to binder ratio causes a significant increase in the adjusted fracture load and modulus of rupture with moderate decrease in permeability. An increase in number of ceramic coats increases the adjusted fracture load significantly and reduces permeability moderately. Impact of the adjusted fracture load on casting shrinkage has been studied by conducting various casting trials. Adjusted fracture load above 315 N is preferred which corresponds to 25–30% silica, filler to binder ratio of 1.25 and number of secondary coats as six to eight for reduced shrinkage variation.

Keywords Investment casting · Slurry composition · Modulus of rupture · Adjusted fracture load · Permeability · Shrinkage

1 Introduction

Originated in 5000 BC, investment casting is one of the most ancient and powerful manufacturing processes. Over the centuries, ceramic shell investment casting process continues to evolve due to advancement in material science technology maintaining sustainable development. This metal casting technique emerges as the most adaptable process to produce high-quality intricate shape, precision casting with an excellent surface finish at comparatively reduced cost. IC process comprises of multiple stages which includes: a production of disposable wax pattern, a formation of ceramic shell to enclose the wax pattern, dewaxing and firing of the ceramic shell and filling, and solidification of the cast metal inside the ceramic shell followed by removal of casting from shell by knockout operation [1]. Ceramic shelling is a unique process which differentiates investment casting from other casting processes. Formation of a ceramic shell is accomplished by repeated dipping of the wax assembly into a refractory slurry of stable viscosity followed by careful rotation of wax assembly at a specific angle which drains and removes the excessive lumps as to achieve uniform coverage and then showering coarse refractory grain over this slurry coat. Hardening of this slurry coat is done by air drying in a humidity-controlled atmosphere. Evaporation of moisture from slurry binder bonds the ceramic stucco particles together to form a coat with refractory particles. After proper drying and hardening of the first coat, all subsequent steps of dipping, draining, showering and drying are repeated until a shell mould of enough thickness is achieved [2].

Ceramic shell mould plays a key role in achieving dimensional accuracy and quality of casting. Majority of investment casting foundries uses zircon ($ZrSiO_4$) slurry

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and zircon stucco for the prime coat which is extremely beneficial. Its fine-grained size provides excellent finish and minute details on surface, the round shape and heavy density improves flowability and drainability offers uniform coat, low coefficient of thermal expansion enhances dimensional stability, its refractoriness offers low reactivity with molten metals, etc. [3]. Secondary and back up coats use other refractory material such as aluminium silicate (Al_2SiO_5), fused silica (SiO_2) or alumina (Al_2O_3) due to cost-effectiveness. The multilayered ceramic mould must possess enough green (unfired) strength to withstand wax removal without cracking or bulging and enough fired strength to withstand the hydrostatic pressure of liquid molten metal. The ceramic shell mould must have high thermal shock resistance to prevent bulging, cracking and leaking during pouring of metal at an elevated temperature. Ceramic shell must have enough permeability to allow escape of air and gases during filling of mould to avoid incomplete mould filling, shell cracking and gas hole defects in casting [4]. Finally, the shell must be easily collapsible after cooling of the metal to facilitate the easy removal of the shell without damage to casting and quick cleaning operation [5]. These requirements can be measured by the modulus of rupture, adjusted fracture load and permeability of the shell in a laboratory and are important properties to ensure above-mentioned mould characteristics. By increasing mould thickness, these properties can be altered, but it results in increased cycle time, high shell material consumption and high labour cost.

In investment casting, the ingredients of slurry play an important role in the characteristics of slurry and properties of a shell. The slurry is composed of refractory flour, binder, water, wetting agent and anti-foaming agents [6]. Various additives such as ceramic fibre, sawdust, rice husk, etc. are also used in slurries to enhance green strength, increase coat thickness, fasten shelling process and improve permeability [7–10]. Typical refractories and binders are major elements used to form the ceramic slurry. The refractory materials (also called as filler), which are used to prepare investment slurries are silica, zirconia, alumina or aluminium silicates [2]. These refractory materials are bound by the binder in the ceramic slurry. The binder solution not only helps the refractory grains in binding but also provides enough strength to the mould. In investment casting, mainly two types of binders are used to form a ceramic shell; ethyl silicate, which is alcohol based and colloidal silica which is a water-based system. Ethyl silicate provides high refractoriness and can be chemically hardened or air hardened quickly, but it provides low bond strength and limited stability in refractory slurries, as compared to colloidal silica. Colloidal silica is the most popular binder used in the precision investment casting industry due to its dimensional stability, chemical inertness and environment friendliness properties [11]. Nowadays colloidal silica is widely used in the industry.

Silica concentration in the binder, as well as filler to binder ratio in slurry are important parameters which can alter the slurry characteristic and affects important mould properties like modulus of rupture, required breaking force and permeability of shell material significantly [12, 13]. High shell strength affects mould dimension prominently in case of a thin-walled hollow components, as it is open completely inside out and restrains free shrinkage of metal. Complex components having multiple dimensions and varying thickness needs multiple feeds to produce sound and shrinkage defect free casting, but the multiple feeds can adversely affect free shrinkage of cast metal causing changes in the dimensions. Initially, the influence of the slurry parameter such as binder concentration, filler to binder ratio and number of ceramic coats on, modulus of rupture (MOR), adjusted fracture load (AFL) and permeability of mould composition has been investigated. Finally, the impact of AFL of mould, on shrinkage of casting has been studied for thin-walled, hollow component which throws light on the dimensional accuracy of these components. The purpose of this study is to establish the relation of slurry composition with shrinkage variation for restricted thin-walled casting.

2 Materials and Methods

2.1 Experiment 1

Initially, the details of slurry composition such as coating details, filler and binder specification have been described to understand the regular shell moulding practice used in investment foundry. Experimental methodology has been presented followed by the procedure to prepare specimen for AFL/MOR and permeability. Methods used to measure the same also have been discussed.

2.1.1 Slurry Composition for Trial

The most common industry practice is to use 30% colloidal silica in the binder and filler to binder ratio as 1.25 for slurry composition. The standard coating sequence used in the trials is as follows: one primary coat of zircon ($ZrSiO_4$) slurry and Midfloor stucco followed by two secondary coats of aluminium silicate (Al_2SiO_5) slurry and stucco of the fine sand grit size of 30:80, followed by four coats of the same with coarser sand grit size of 15:30 followed by the final seal coat. Details of these coats are provided in Table 1. The secondary slurry composition is provided in Table 2 and binder specification is given in Table 3.

Table 1 Specification standard for shell coating sequence

Coats	Refractory material	No. of coats	Stucco grit size	Binder (colloidal silica)	Filler grit size	Viscosity (s)	Humidity (%)	Drying time (hours)
Primary	Zircon	1	< 100	30%	− 200	22–24	40–60%	12
Secondary	Aluminium silicate	2	30:80		− 300	16–18		6
Backup		4	16:30					

Table 2 Filler % specification: Aluminium silicate

SiO_2	55.0%
Al_2O_3	42.0%
TiO_2	1.5%
Fe_2O_3	1.5%
Density	2.2%

Table 3 Binder specification: silica (SiO_2)

Concentration	30%
pH at 30 °C	9.5–10.5
Titrable alkali-Na_2O	0.6
Chlorates/sulphates	Traces
Density at 25 °C	1200 kg/m^3

Figure 1 shows the methodology of experiments. Impact of slurry parameters on mould properties has been investigated in experiment 1, whereas influence of these parameters on shrinkage of investment casting is explained in experiment 2. The details of experimental design are shown in Table 4. Preparation of specimens for experimental work has been discussed in next section.

2.1.2 *Preparation of Specimens for MOR, AFL, and Permeability Test*

Rectangular wax plates having dimensions 100 × 25 × 6 mm as shown in Fig. 2 have been used to measure the MOR and AFL of mould. Specimens were prepared by dipping the wax plates repeatedly in various ceramic slurries, as per experimental design shown in Table 4. This was followed by draining, stuccoing and air

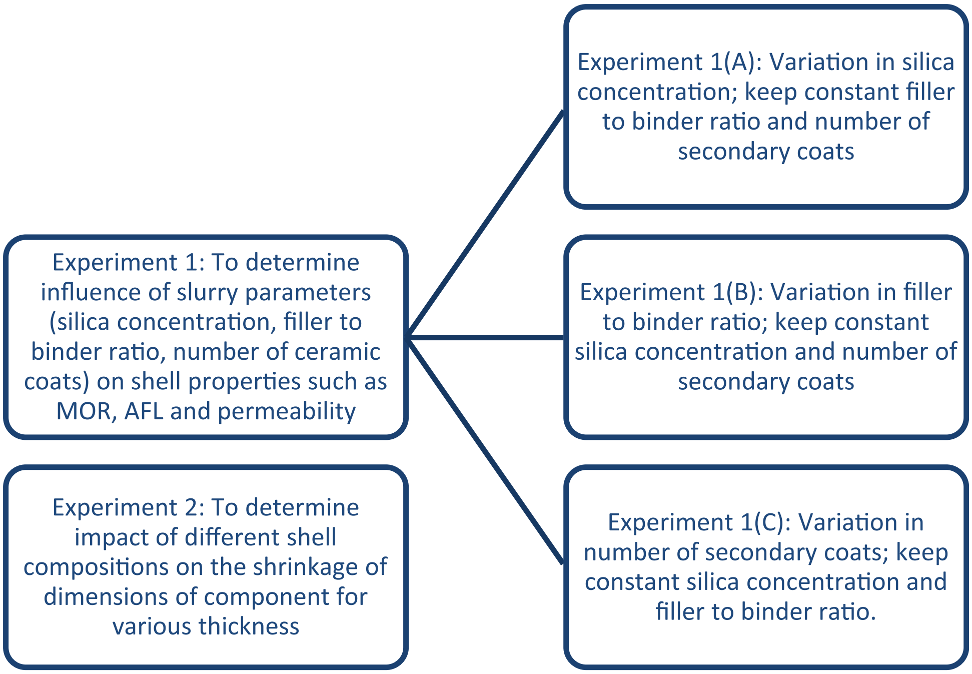


Fig. 1 Experimental methodology

Experiment 1: To determine influence of slurry parameters (silica concentration, filler to binder ratio, number of ceramic coats) on shell properties such as MOR, AFL and permeability

Experiment 1(A): Variation in silica concentration; keep constant filler to binder ratio and number of secondary coats

Experiment 1(B): Variation in filler to binder ratio; keep constant silica concentration and number of secondary coats

Experiment 1(C): Variation in number of secondary coats; keep constant silica concentration and filler to binder ratio.

Experiment 2: To determine impact of different shell compositions on the shrinkage of dimensions of component for various thickness

Table 4 Experimental design to check impact of mould parameters on mould properties

	Silica concentration	Filler to binder ratio	Number of ceramic coats
Experiment 1(A)	Varies (20%, 22.5%, 25%, 27.5%, 30%)	Constant (1.25)	Constant (6)
Experiment 1(B)	Constant (30%)	Varies (0.75, 0.875, 1, 1.125, 1.25)	Constant (6)
Experiment 1(C)	Constant (30%)	Constant (1.25)	Varies (4, 5, 6, 7, 8, 9)

Fig. 2 Test piece for modulus of rupture

Fig. 3 Test piece for permeability

drying in humidity-controlled atmosphere as mentioned in Table 1. Similarly, the test pieces for permeability test were also prepared for all three types A, B, and C of the ceramic composition. Plastic table tennis balls were pierced at one end by inserting the long glass tube as shown in Fig. 3 which was then dipped into ceramic slurries of different compositions followed by drying. Shell specimens were sent to furnace for dewaxing and are sintered at 900 °C for 2 h. Hot permeability was measured for each shell made from different slurry compositions as per experimental design mentioned in Table 4.

2.1.3 Measurement of Modulus of Rupture (MOR)

In investment casting process, during preheating and pouring, the thin ceramic mould has to withstand extreme high temperature; thus, mould strength check is essential to avoid cracking, leaking and bulging of mould. Compressive strength of ceramic is much higher than the tensile strength; a flexural bending test is most commonly preferred as a measure of mould strength. A modulus of rupture (MOR) test is one of the measures of flexural bending strengths which give an indication of how many coating layers will be required with each sample composition. In this research work, the MOR of the samples was determined through three-point flexural testing.

Ceramic strips of standard dimensions length (*L*), width (*W*) and thickness (*H*) were prepared by coating wax strips for different ceramic compositions. These specimens were tested for MOR of a shell by using universal strength testing machine where the three-point test was carried out on ceramic plate specimen. In this test a flat, rectangular specimen is simply supported near its ends (75 mm apart) by knife edge support resting on a smooth surface of the specimen. The other rough surface is loaded centrally on top as shown in Fig. 4. The applied load was increased in steps till the shell specimen broke and the breaking load (*F*) was recorded. The test was conducted on the green stage, i.e. at room temperature as well as for the sintered shells at an elevated temperature which was close to actual working condition. The MOR was calculated using Eq. 1 on a green specimen and on a sintered specimen

$$\text{Modulus of rupture MOR }(\text{N/mm}^2) = \text{MOR} = 3\text{FL}/2\text{BH}^2 \tag{1}$$

where *L* distance between two resting point (75 mm constant), *B* width of specimen in mm, *F* breaking load in kg, *H* thickness of bar at breaking point.

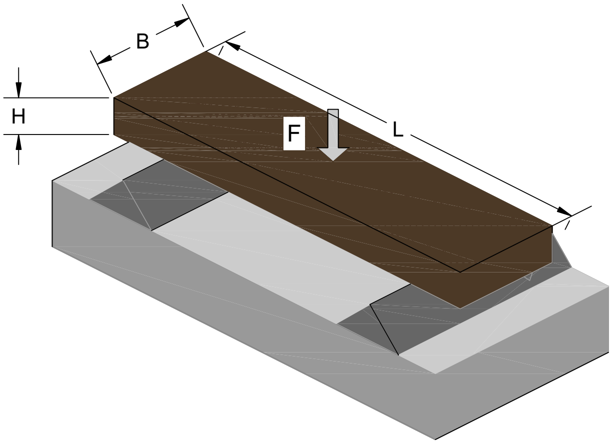


Fig. 4 Set-up for measurement for modulus of rupture test

2.1.4 *Measurement of Adjusted Fracture Load (AFL)*

Another parameter of interest for flextural testing of ceramics is adjusted fracture load (AFL). Thickness (*H*) of investment casting mould is a function of slurry and stucco parameters such as number of slurry and stucco coats, slurry viscosity and size of stucco. By multiplying the flextural stress of test sample by their thickness squared, the load capacity of specimen is calculated

$$\text{Adjusted fracture load } (N) = \text{AFL} = f * \text{MOR.H}^2 \qquad (2)$$

where *f* is a constant factor and *H* thickness of part.

The averages of the measured AFL values were adjusted using the constant factor '*f* 'which was set to the lowest value of 1. Calculated AFL values using Eq. 2 for the samples showed the results similar to flexural stress. The AFL directly relates to the performance characteristics of interest during casting and pattern removal. AFL is of use when the structure of a tested material is not uniform throughout. '*f*' *is* a constant factor used to normalize the AFL value, while the unit for AFL is same as force, i.e. Newton but it does not have a direct physical meaning. The AFL value is a reference for comparing relative load capacities between multiple samples. This makes it easier to compare differently processed moulds in regards to load carrying capacity during pattern removal and pouring.

2.1.5 *Permeability Measurement*

Tennis balls with a long glass tube pierced at one end have been used as a specimen for permeability measurement. As per experimental design, a number of slurry layers of different slurry composition have been built on the tennis ball to form ceramic balls (bulbs) followed by drying operation. These ceramic shells with the plastic ball inside have been heated in the furnace where the plastic balls burnout. Hot pressurized steam was used to clean the ceramic bulbs from inside. The specimen was then connected to the permeability tester with a hose pipe as shown in Fig. 5. A constant air flow was regulated by a valve and measured by a flow meter. Air was passed through the ceramic bulb and the back pressure generated due to the permeability of the ceramic ball was measured by the pressure gauge connected in the circuit. The permeability of the shell was measured when the shell was at room temperature as well at the sintering temperature in the furnace at 900 °C using Eq. 3

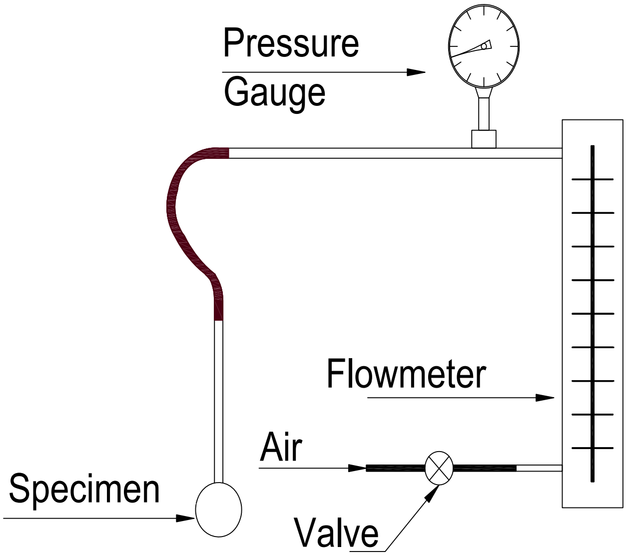


Fig. 5 Set-up for permeability measurement

$$\text{Permeability}(k) = {}^{(Q * \mu * T)}\!/\!_{[(A * (\Delta P)]} \qquad (3)$$

where k permeability in cm^2, Q air flow rate in cm^3/sec, μ dynamic viscosity of air N-s/m^2, T thickness of shell in cm, A surface area of inner wall of ball in cm^2, ΔP pressure difference in N/m$^{2.}$

2.2 Experiment 2

In the second experiment of the research work, nine combinations of shelling systems were selected for the experiment with varying values of AFL (lowest to highest) and their impact on variation of casting shrinkage was investigated by conducting casting trials. The description of component used for this experiment and shrinkage calculations has been described in following sections.

2.2.1 *Component Description*

A rectangular shaped, thin-walled, hollow component has been selected for casting trial as shown in Fig. 6. This component is considered as one of the most complex geometries since it needs multiple feeds to produce sound

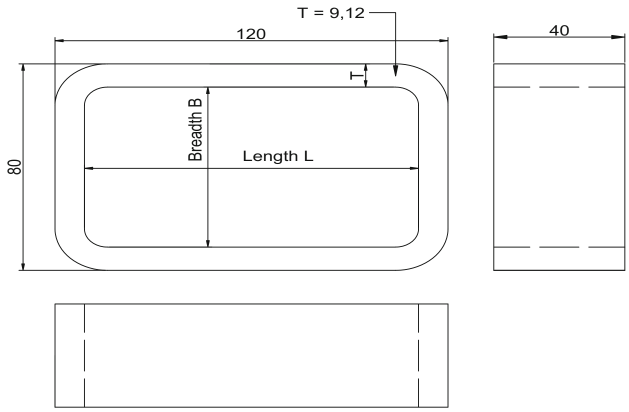


Fig. 6 Drawing of selected specimen (Dimensions in mm)

Fig. 7 Shell mould for component

casting. Using simulation software, the feed size and location were determined. Wax patterns along with designed feed were produced in water cooled CNC milled aluminium die. Two critical dimensions of the rectangular cavity, inside width ($W = 56$ and $W = 62$ mm) and length ($L = 96$ and $L = 102$ mm) were measured on two different wall thickness, $T = 9$ and $T = 12$ mm, respectively. As depicted in Fig. 7, the eight-wax tree was assembled by attaching two-wax pattern of two different thicknesses on each face of the vertical riser plate with sprue at one end and test piece at other end.

2.2.2 *Mould Trials & Shrinkage Calculations*

All wax tree assemblies were dipped into slurries corresponding to different combinations chosen as per their AFL values. After drying, dried ceramic moulds were dewaxed in a furnace for cavity formation and then sintered for achieving proper strength. At the pouring temperature of 1550 °C, austenitic stainless steel was immediately poured in the newly formed cavity. After solidification and cooling, the shell was removed by knockout operation and the castings were then separated from mould by cutting saw or grinding wheel. Critical dimensions were measured on finished castings. Shrinkage was calculated from wax and casting dimensions using Eq. 4 which was then plotted against corresponding AFL of mould composition

$$\%\text{Shrinkage} = 100\,(L_{\mathrm{w}} - L_{\mathrm{C}})/L_{\mathrm{w}} \qquad (4)$$

where L_{w} is measurement on wax patterns and L_{C} is measurement on casting.

3 Results and Discussion

Various experimental results obtained by varying slurry parameters on mould properties have been discussed herewith.

3.1 Influence of Silica Concentration on Mould Properties

Figure 8 shows the trend of modulus of rupture (MOR) and adjusted fracture load (AFL) against varying percentages of the silica. Observations reveal that MOR and AFL of shell at the fired stage is higher than the green shell strength by approximately 60% for each step change in silica concentration. Fired breaking force increases linearly with increasing concentration factor up to 27.5% and thereafter gets stagnated. This proves and confirms that 30% concentration of silica used is optimum for industrial applications. As the percentage of silica concentration in alkaline solution (Ph value 9.5–10.5) increases, positively charged (Na+) sodium particles repel each other thereby increasing the surface area of silica particles. This improves the enveloping of filler particles further increasing the strength of bond between particles. Evaporation of water vapour reduces OH- concentration and converts Na+ charged particles to stable salts. The size of colloidal silica particles in the solution has been controlled by adjusting the pH and sodium concentration. As the Ph value is reduced below 8.3, silica molecule will come close and polymerization will start leading to gel formation with stable salts formed earlier. Hence, higher the silica concentration (up to 27%), higher the gel formation which will result in higher AFL of the shell. Figure 9 reports that there is marginal increase in fired permeability at lower level of silica from 20% to 25%, but no significant change in permeability is observed thereafter.

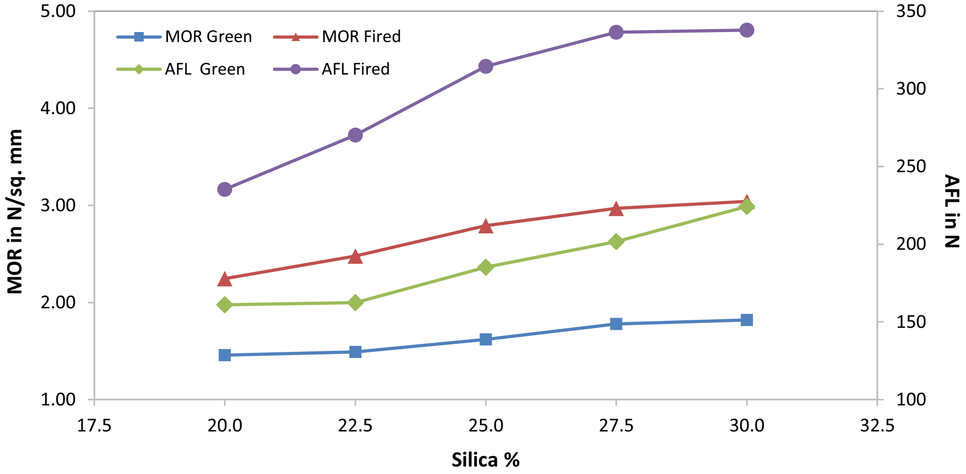


Fig. 8 Influence of silica concentration on MOR and AFL

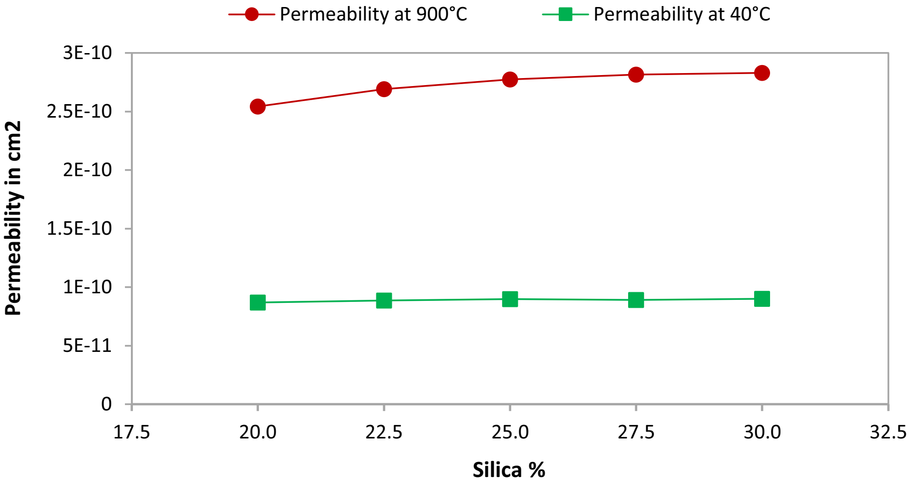


Fig. 9 Influence of silica concentration on permeability

3.2 Influence of Filler to Binder Ratio on Mould Properties

In these experiments, silica concentration has been kept constant at 30% and the number of coats at six. The effect of changes in filler to binder ratio from 0.75 to 1.25 on MOR, breaking force (AFL) and permeability has been observed. Figure 10 depicts that as the filler in the slurry increases, the MOR and breaking force (AFL) in green stage as well as in the fired stage also increases. The actual permeability in the green stage as well in the fired stage shows decreasing trend for increasing filler concentration as depicted in Fig. 11. An increase in filler to binder ratio makes the slurry more viscous, resulting in thicker coating of slurry after every dipping process and thus more sand particles get embedded in every coat during stuccoing. This results in increased coat thickness and thus in increased growth for MOR and breaking force. However, a thicker slurry finds difficulty in draining out and reaching minute corners of intricate casting. Thus, industry practice is to keep the ratio at 1 to 1.25 maximum. As the filler to binder ratio in shell increases, the shell becomes less porous and thus permeability reduces. The particle size of the filler particle is very fine (< 200 mesh), whereas sand used is coarser. Increase in filler to binder ratio increases the presence of fine particles in the shell, thus increases the resistance to flow and decreases the permeability of the shell.

3.3 Influence of Number of Ceramic Coats on Mould Properties

In this trial, silica concentration is kept constant at 30%, and filler to binder ratio equal to 1:1. Various experiments have been conducted by changing the number of secondary ceramic coats between 4 and 10. The effect of the number of ceramic coats on MOR and AFL has been indicted in Fig. 12. It is observed that as the number of coats increases from 4 to 10, there is a slight increase in AFL at green stage but the increase in fired AFL is significant. As the number

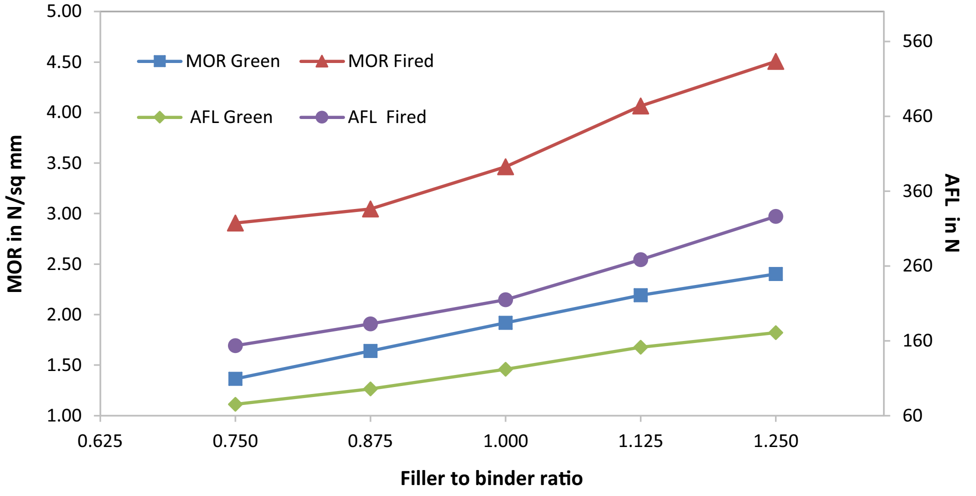


Fig. 10 Influence of filler to binder ratio on MOR and AFL

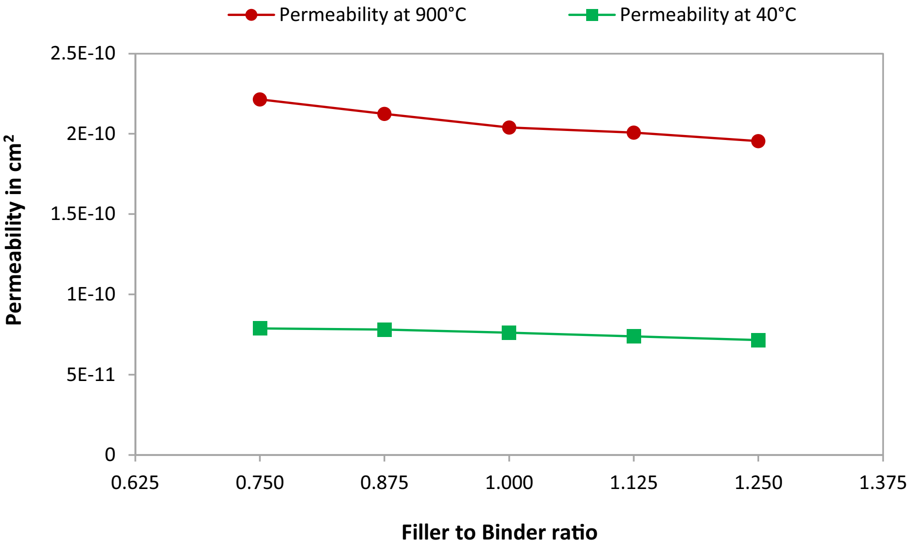


Fig. 11 Influence of filler to binder ratio on permeability

of coats increases, shell thickness increases which results in growth in AFL. Also, AFL increases exponentially as the number of coats increases above eight. Since the only change in the composition of the shell is increase in shell thickness, the MOR is constant. Figure 13 depicts that, as the number of coats increases, thickness of the shell increases causing more restriction to air flow and thus the slight decrease in permeability. Change in grain size of the filler sand particle is the most effective parameter to achieve significant impact on permeability.

3.4 Influence of AFL on Casting Shrinkage

All the above experiments conclude that MOR and AFL of shell increases with growing concentration of silica in the binder as well as the increase in filler to binder ratio. Mould trial with varying AFL values from highest to lowest level has been selected as shown in Table 5. Casting trials have been conducted to observe the impact of AFL on shrinkage of critical dimensions of the selected component. The results are shown in Figs. 14 and 15.

This indicates that as the AFL of mould increases, percentage shrinkage of both the critical dimensions, i.e. length as well as breadth decreases, and the trend is exponential in nature. This indicates that as the mould becomes stronger thus less shrinkage/contraction is observed in the component. The selected component have a hollow shape and is surrounded by mould inside out thus offering constraint from all sides of the component. Lower AFL is suitable for mould due to its ease in shell removal, however it results in a weak shell and may lead to leakages during filling and solidification. The trend line of shrinkage become flatter above AFL values of 315 N and consistent geometrical stability is observed for the shell composition which gives AFL values above 315 N. Hence, the combination of silica content 25–30%, Binder to filler ratio as

Fig. 12 Influence of number of ceramic coats on MOR and AFL

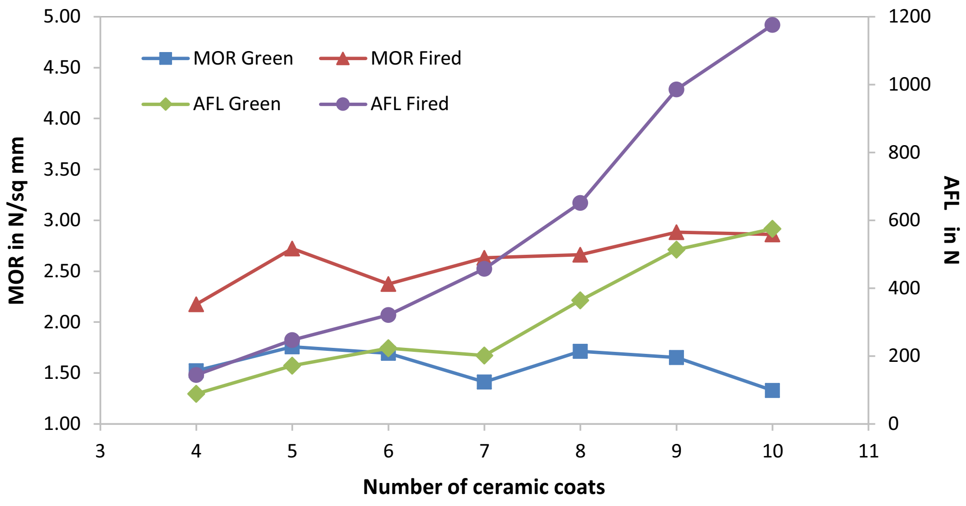


Fig. 13 Influence of number of ceramic coats on permeability

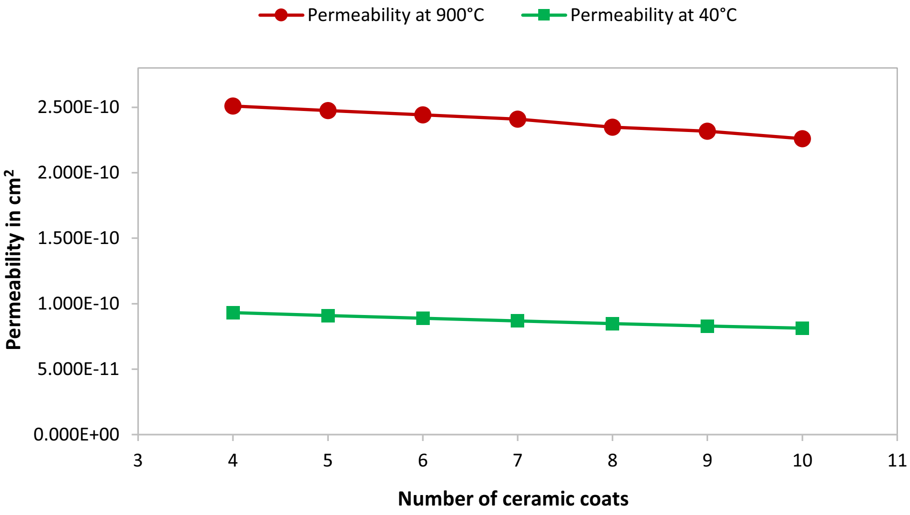


Table 5 Selected slurry parameter for varying AFL values

Adjusted fracture load	Silica %	Filler to binder ratio	Number of ceramic coats
153	30	0.75	6
183	30	0.875	6
215	30	1	6
235	20	1.25	6
269	30	1.125	6
315	25	1.25	6
326	30	1.25	6
336	27.5	1.25	6
457	30	1.25	7
652	30	1.25	8

1.25 and number of coats between 6 and 8 are better choice for such a rectangular shaped component having weight in the range of 1–3 kg.

Observations also indicate that the part with higher thickness (12 mm) shows larger shrinkage compared to the part with smaller thickness (9 mm). As per Chvorinov's rule, the casting with a thin wall, i.e. having a large surface area and small volume will cool rapidly than a casting with a small surface area and large volume. Thus, thick (12 mm) section causes more shrinkage compared to the thin section (9 mm). Comparative study of shrinkages of both the dimensions shows that less shrinkage is observed in length as compared to width. Keen observation of mould assembly reveals that the selected component needs multiple feeds for sound casting due to its thin wall and a hollow shape. Total four feeds, two on each long side of the component have been attached which constrains the free shrinkage of height compared to width. Increase in the number of ceramic coats from 6 to 10 increases the strength of mould multifold which require an extremely high load to break the shell and to clean the casting. All the casting has

Fig. 14 Influence of AFL on breadth shrinkage

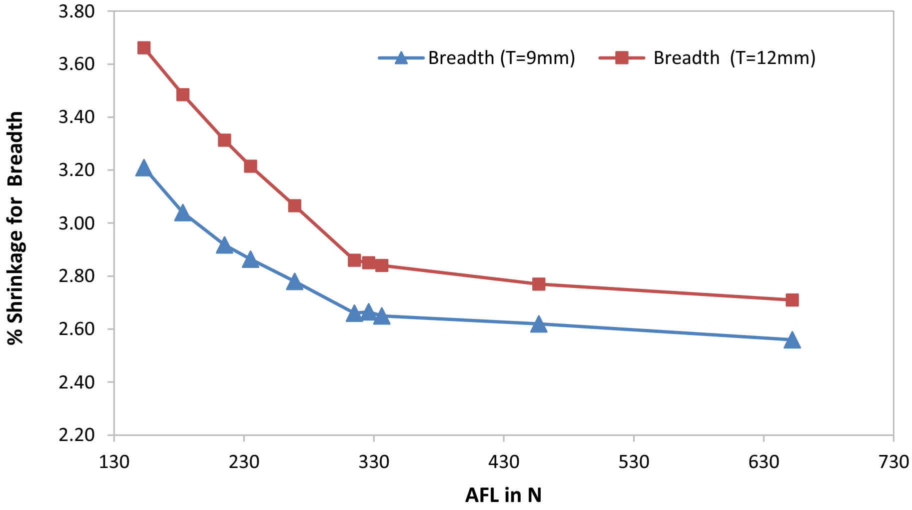
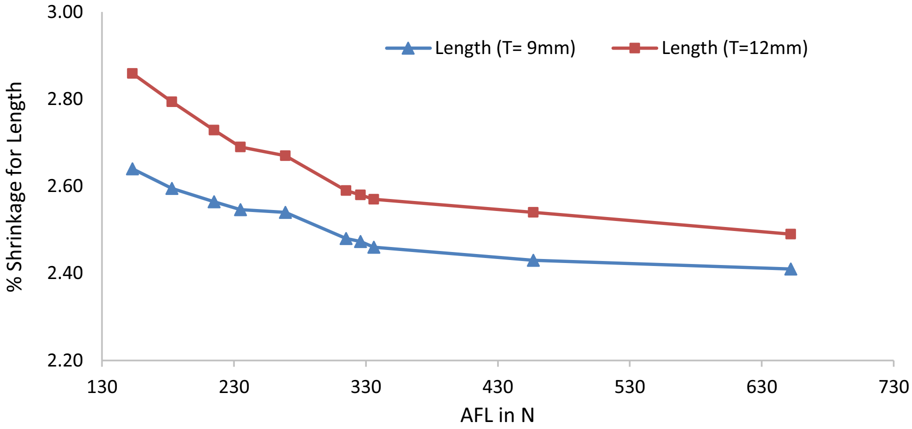


Fig. 15 Influence of AFL on length shrinkage



been tested for gas hole defect due to lower permeability using radiography testing and is found to be ok.

4 Conclusions

In this work, the effect of slurry parameters such as binder concentration, filler to binder ratio and number of ceramic coats on mechanical properties of ceramic mould, namely MOR, AFL and permeability have been investigated by conducting a series of experiments. As shrinkage affects dimensional accuracy, the relationship between AFL and cast metal shrinkage has also been established. Selected slurry parameters have significant effect on mould properties as discussed below.

- The experimental results indicate that as the percentage concentration of colloidal silica binder in ceramic slurry increases, the AFL increases up to 27.5% and then becomes stagnant. Further increase in silica concentration does not improve AFL. Thus, it can be concluded that 27.5–30% of colloidal silica is the optimum value.
- Increase in filler to binder ratio causes increase in viscosity of slurry which attracts more stucco particles to get embedded in each coat. This results in increase in strength significantly but the filler to binder ratio is optimized at 1.125–1.5 which eases draining operation as well as reaching complicated corners of the component.
- As number of coats applied on ceramic mould increases, AFL intensifies. Observation concludes that AFL is suddenly increased after 8 coats which is not suitable since it finds difficulty during knockout operations. The permeability reduces as the number of coats increase.
- Increase in AFL of mould restrains the free shrinkage of alloy, thus a decrease in casting shrinkage is observed. AFL values above 315 N provide dimensional stability as shrinkage variation is negligible. Thus, selecting silica content as 30%, binder to filler ratio as 1.25 and number of coats between six and eight,

provides the optimized combination of slurry parameters for dimensional stability. Feeder constraint dimensions (breadth) has more restrain for the free shrinkage compared to non-constraint dimensions (length). Thick component experience more shrinkage compared to thin component.

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