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# Investigation of fibre modified ceramic moulds for investment casting

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#### Abstract

The incorporation of nylon fibres in a water based silica binder to replace the costly liquid polymer addition in investment casting mould were investigated in this work. Ceramic moulds produced with fiber modified binder were found to be 13% thicker at flat section and 40% thicker at sharp edges and radii than polymer modified system. The firing strength after fiber and polymer burn out was comparable for both systems. The extra shell thickness in the fibre system, especially at edges, gives a higher load bear capacity. This has the advantage of resisting cracking during de-wax and mould wall movement during alloy casting which lead to costly defects. A much higher mould permeability at casting temperature of fibre modified system also proves advantageous to complete mould filling. The unique properties of fibre modified system lead to a promising alternative for the standard investment casting industry. © 2002 Elsevier Science Ltd. All rights reserved.

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# 1. Introduction

Investment casting allows dimensionally accurate components to be produced in high or low volumes-a much cheaper alternative to forging or metal turning since the waste material is kept to a minimum.<sup>1</sup> The production of the investment casting ceramic mould is a crucial part of the whole process and can be summarized as follows.<sup>2,3</sup> First, multi-component slurries are prepared, which normally consist of a refractory filler and a binder system. A pattern wax is dipped into the facecoat slurry, sprinkled with a coarse grained refractory stucco and dried. The binder coalesces during drying to form a gel network, which binds the ceramic material together. The dipping process is repeated many times to produce a graded mould. Flexibility exists in changing the composition of each layer. The first or 'primary' coat applied to the wax will ultimately be in contact with molten alloy, and its composition therefore often differs from that of secondary or 'backup' coats. The wax pattern is removed, normally by high pressure steam autoclave, leaving a hollow mould. Moulds are

fired and cast with molten metal. After cooling, the ceramic is removed by mechanical or chemical methods.

The exacting requirements of an investment casting mould include: I. Sufficient green (unfired) strength to withstand wax removal without failure; II. Sufficient fired strength to withstand the weight of cast metal; III. High thermal shock resistance to prevent cracking during metal pouring; IV. High chemical stability to prevent the mould-metal interaction; V. Sufficient mould permeability to allow trapped air permeate through the mould walls during metal pouring; VI. Sufficient thermal conductivity to maintain an adequate thermal transfer through the mould wall and hence allow the metal to cool and VII. Limited creep to prevent dimensional changes within the mould wall and ultimately the casting.<sup>1,4</sup>

Various combinations of materials have been used to produce the ceramic mould, but most investment foundries use a silica based binder system. These binders can be either water or alcohol based. Following the introduction of the Environmental Protection Act in 1992, the degree of environmental emissions allowed from industrial processes has been limited. This has resulted in increased use of water-based binders which reduce volatile organic compound (VOC) emissions. Colloidal binders produce ceramic moulds with very low green strengths which are prone to cracking during wax

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removal and handling.<sup>5</sup> This is overcome by the use of liquid polymer additions, either latex based for alkaline systems or polyvinyl alcohol (PVA) based for acidic system. Polymers increase the ceramic green strength and have a tendency to reduce the level of "wet-back" of moisture into previously dried coats when new layers are added.<sup>6</sup> A unique feature of the colloidal silica binder is that it can convert from a liquid to a semi-solid by gellation, which is the means by which the slurry layers are bound. The coalesced binder eventually converts to silica, which is a refractory in its own right. However, production of the large moulds using conventional silica bonded ceramics has often resulted in unacceptable casting failure rates. One of the main causes of the failure has been attributed to casting bulge. Casting bulge involves large areas of external casting surface being convex instead of flat and is thought to result from mould wall movement after the pouring of the metal but prior to metal solidification. Bulging can be alleviated by adding extra coats to produce thicker mould walls. However, this process is labor intensive, time consuming, and therefore costly for the foundry.

One recently patented method for reducing shell production times and enhancement of ceramic mould properties is the use of organic fibre additions into ceramic slurries<sup>4,7</sup> thus negating the need for expensive liquid polymer additions. This investigation involved a comparison of a polymer modified mould system and a fiber modified slurry system, which produces a thicker shell without adding extra coats. Properties such as modulus of the rupture (MOR), adjusted fracture load (AFL), strength of the trailing edge and shell permeability were studied. The MOR, measured in the 3-point bend mode is an intrinsic material property unaffected by the dimension of the test bar. The AFL takes the shell thickness into account and therefore represents the actual shell performance when the shell is subjected to an external load.<sup>4,8</sup> The MOR test method has been critically examined by Okhusen and Voigt<sup>9</sup> and has been widely used in industry, particularly to promote the properties of one mould material over another.<sup>10,11</sup> However, the most frequent site of mould failure during dewax and casting is at sharp radii and corners,<sup>12</sup> where the coverage of slurry and stucco will be critical. The MOR test does not determine the ability of the mould to resist cracking in these areas. In this paper, a wedge test developed by Leyland etc.<sup>12</sup> was used to evaluate the strength and load capacity at edges and corners. This was then related to the success of the mould during casting. As higher permeability is an advantage for moulds during casting, the permeability of the shell systems was also measured and presented in this paper. The results show that the fibre modified system processes a range of unique properties, leading to enhanced shell build and the possibility of using fewer coats for equivalent ceramic thickness and mechanical performance.

# 2. Experimental

#### 2.1. Shell production

Ceramic moulds with zircon/silica facecoats were produced. Details of slurry composition and mould build are listed in Tables 1 and 2. The primary slurry consisted of a colloidal silica binder (Remet, LP-BV) and zircon filler (Johnson Mathey,-200 mesh Zircosil). A predetermined amount of liquid polymer (Remet, Adbond ADII), wetting agent (Remet, Victawet 12) and anti-foam (Remet, Burst RSD-10) were added to the colloidal silica sol before filler addition. The properties of the resultant slurry were characterized by measurements of density, pH and viscosity. The viscosity was determined by the time required for a definite volume of slurry to flow through the orifice in the bottom of a metal flow cup (B4 cup), and the density of measured using pyknometer (100 ml, Sheen 1501) with a two decimal place electron digital balance.

The standard polymer modified backup slurry contained 6 wt% polymer. The fiber modified backup slurry contained nylon fiber (Wex Chemicals Ltd., Wex-Perm<sup>TM</sup>) with a ratio of 20 g per litre of total liquid. The shells were made by first investing the wax pattern into the primary slurry. An alumino-silicate stucco (ECC International, Molochite 50/80 mesh) was applied by the rainfall sanding method. The coat was dried at a temperature of 21 °C, 50% relative humidity and 0.4 m/ s air speed for 24 h. Four backup coats were then applied. A coarse alumino-silicate stucco (ECC International, Molochite 30/80 mesh) was used as a backup stucco. Each secondary coat was dried at a temperature of 21 °C, 50% relative humidity and 3m/s air speed for 90 minutes. Finally a seal coat of secondary slurry was applied and dried at a temperature of 21 °C, 50% relative humidity and 3 m/s air speed for 24 h. The wax inside the ceramic mould was then removed by steam autoclaving at 8 bar pressure for 4 min, followed by a controlled de-pressurisation cycle at 1 bar/minute using a Quicklock Boilerclave<sup>TM</sup> (Leeds and Bradford Boiler Company Ltd., UK).

For MOR testing, the samples were prepared upon a wax pattern with dimensions of 200 mm $\times$ 25 mm $\times$ 10 mm thickness. After dewax, the moulds were cut into rectangular test bars. For the edge testing, the test pieces were taken from moulds produced using a specially designed wax pattern, as shown as in Fig. 1, which produces symmetric trailing edge sections. The length of the edge test sample was approximately 20 mm and the width of the sample 10 mm. To simulate shell firing conditions, the samples were fired at 1000 °C for 60 min.

For the measurement of permeability, a test piece former, as shown as in Fig. 2. was prepared. The mullite ceramic tube (6 mm internal diameter and 10 mm outer diameter and 250 mm in length) penetrates into the

 Table 1

 Slurry compositions for ceramic mould samples

Slurry	Materials	Composition
Primary	LP-BV silica sol <sup>a</sup>	25% silica
	Adbond ADII polymer <sup>a</sup>	6 wt.% of silica sol
	Victawet 12 wetting agent <sup>a</sup>	0.3 wt.% of total liquids
	Burst RSD-10 antifoam <sup>a</sup>	0.5 wt.% of total liquids
	-200 mesh Zircosil <sup>b</sup>	77% refractory loading
Secondary (Polymer modified)	LP-BV silica sol <sup>a</sup>	25% silica
	Adbond ADII polymer <sup>a</sup>	6 wt.% of silica sol
	-200 mesh Alumino-silica <sup>a</sup>	57% refractory loading
Secondary (Fibre modified)	silica sol 1030°	25% silica
	WexPerm <sup>TM</sup> fiber <sup>c</sup>	20 g fiber per litre of liquids
	-200 mesh Alumino-silica <sup>a</sup>	57% refractory loading

<sup>a</sup> Remet, UK.

<sup>b</sup> Cookson Mathey Ceramics Ltd.

° Wex Chemical Ltd.

Table 2Shell build for polymer modified and fiber modified samples

Coat	Slurry type	Stucco	Dip time (s)	Drain time (s)	Dry time (h)
1	Primary	50/80 M	30	60	24
2–5	Secondary	30/80 M	30	60	1.5
6	Secondary	-	30	60	24

 $M = Molochite^{TM}$  (alumina-silicate); 30/80 = 30/80 mesh stucco; 50/80 = 50/80 mesh stucco.

hollow plastics ball (20 mm radius) between 5 and 20 mm. A small fillet of wax was used to bond the tube to the ball and act as a sealant. The test pieces were then dipped to produce shell samples. The shells were slowly heated up to 700 °C at a rate of 3 °C/min in a furnace which has a extraction system at room temperature. The shells were held at 700 °C for 5 min to completely remove any trace of the table tennis ball, and then heated up to 1000 °C for 60 min to fire.

#### 2.2. Properties measurement

To evaluate the mechanical properties of the moulds, three-point bend tests were conducted on the green and fired bars at room temperature. Samples were loaded in an Instron 8500 tensile testing machine at a constant load rate of 1 mm/minute until failure. All samples taken into account broke at or near the center, so that no correction was necessary to allow for stress gradient in the span. The MOR,  $\sigma_{Max}$ , was calculated using Eq. (1)

$$\sigma_{\text{Max}} = \frac{3P_{\text{Max}}L}{2WH^2} \tag{1}$$

where  $P_{\text{Max}}$  is the fracture load, W and H are the width and thickness of sample fracture area, L is the span length. The load/deflection curve was converted into a stress/strain curve using Eqs. (2) and (3).

$$\sigma = \frac{3PL}{2WH^2} \tag{2}$$

$$\varepsilon = \frac{6H\delta}{L^2} \tag{3}$$

where  $\sigma$  is the stress,  $\varepsilon$  is the strain,  $\delta$  is the deflection and *P* is the corresponding load.

Adjusted fracture load in bending  $(AFL_B)$ , defined as the load necessary to break a 10 mm wide shell test piece across a 70 mm span, normalises the load bearing capacity of the shell and can be expressed as Eq. (4)



Fig. 1. (a) The wax pattern for edge test; (b) the configuration of the wax pattern section l; and (c) the configuration of the ceramic edge test piece.



Fig. 2. Schematic section through permeability test piece coated with ceramic shell.

$$AFL_{\rm B} = f_{\rm B}\sigma_{\rm Max}H^2 \tag{4}$$

where  $f_{\rm B}$  is a constant equal to 0.1.

For the edge test, instead of testing a plane mould surface, a wedge was forced into the \*test piece (see Fig. 3). The test piece was loaded such that the inner surface of the mould (the primary layer) was in tension and the outer surface in compression. The load required to break the test piece was recorded and the strength of the edge piece was calculated using Eq. (5) [13]

$$\sigma_{\text{wedge}} = 12.2 \frac{\sin\theta\cos\theta Fd}{WT^2} \tag{5}$$

where F is the fracture load applied to the wedge, d is the span length, W is the width and T is the thickness of edge test piece (see Fig. 3).

Adjusted fracture load of the edge sample  $(AFL_W)$ , defined as the load necessary to break a 10 mm wide edge test piece with a 20 mm span length, normalises the load bearing capacity of the shell at edges and can be expressed as Eq. (6)

$$AFL_{\rm W} = f_{\rm W}\sigma_{\rm Wedge}T^2 \tag{6}$$

where  $f_{\rm W}$  is a constant equal to 0.17.

The permeability of the shell was determined by recording the flow of air through a shell mould of known dimensions under a known pressure difference and at a given temperature (BS 1902: section 10.2:1994). Samples were heated up at a rate of 20 °C/min and held at the designed test temperature for 12 min. The pressure difference and the flow were recorded at 5 and 10 min dwell times. The permeability of the shell,  $\mu$  (in m<sup>2</sup>), was calculated using Eqs. (7) and (8).



Fig. 3. The schematic loading configuration for the wedge test.

$$\mu = \frac{\eta V_c l}{aP} \tag{7}$$

with

$$V_{\rm c} = \frac{V_1 T}{T_1} \tag{8}$$

where

- $\eta$  is the dynamic viscosity of air at the temperature of the test (in Ns/m<sup>2</sup>)
- $V_{\rm c}$  is the volumetric flowrate of air corrected for expansion at elevated temperatures (in m<sup>3</sup>)
- $V_1$  is the volumetric flowrate of air at room temperatures (in m<sup>3</sup>)
- l is the thickness of the shell mould (in m)
- *a* is the inner surface of the hollow shell mould (in m)
- P is the pressure difference across the test piece (in N/m<sup>2</sup>)
- T is the elevated temperature (in Kelvin)
- $T_1$  is the room temperature (in Kelvin).

# 2.3. Microstructural observation

Scanning electron microscopy (SEM) and optical microscopy were used to observe the microstructure of the ceramic shell and the fiber morphology. Samples observed under the SEM were sputter coated with either gold or carbon to form a conducting layer to avoid electrical charging.

# 3. Results and discussion

### 3.1. Fiber morphology

The morphology of the fiber, observed by SEM, as shown as in Fig. 4. The average length of fiber is approximately 1 mm, with a diameter of 20  $\mu$ m. The surface of the fiber is quite smooth, which will have implications for the relative ease of fibre pull-out during the application of mechanical force.





Fig. 4. Secondary electron microscopy image showing morphology of acrylic nylon fiber at different magnifications.

#### 3.2. Slurry properties

The physical properties of the primary slurry and the two secondary slurries were measured and listed in Table 3. The main slurry properties of two backup slurries such as viscosity, pH and the density were identical. During shell production, however, fiber modified shells had a much reduced drain time. This gave a higher ceramic retention rate after dipping and led to a thicker coat compared to that produced with polymer modified slurry. This could be related to the relative surface tension between the fiber surface and liquid, but further study will be carried on to quantify the mechanism.

#### 3.3. Shell properties

#### 3.3.1. Shell build

Comparison of the ceramic shell thickness achieved for polymer modified moulds and fiber modified moulds is listed in Table 4. Little shrinkage was observed after firing of the samples. Thinner mould walls at the edges were found in both systems, see Fig. 5, showing reduced coverage and shell build at this point. It has been reported that most mould failures occur along the entire length of the trailing edge[12]. With identical material and shell build condition, the fiber modified shells were 13% thicker at flat section than polymer modified shells over a six-coat system. At the trailing edge, the fiber modified shells were 40% thicker than polymer modified shells. Therefore, to produce an equivalent shell thickness at the flat section, the number of secondary coats can be reduced by using fibre modified slurry, which still gives better coverage at the sharp corner. This has

Table 3 Comparison of slurry properties

Slurry	pН	Viscosity (s) B4 cup	Density (kg/m3)
Primary Secondary (polymer modified)	9.4–9.8 9.4–9.8	100–120 28–30	2.84
Secondary (fiber modified)	9.4–9.8	28-30	1.61

Table 4 Comparison of the mould thickness

Mould sample	Status	Thickness of the flat piece (mm)	Thickness of the trailing edge (mm)
Polymer modified mould	Green	4.6	2.5
	Fired	4.6	2.4
Fiber modified mould	Green	5.3	3.5
	Fired	5.2	3.5



Fig. 5. Comparison of the thickness of the shell. (a) polymer modified shell; (b) fiber modified shells.

significant reduce on production time and material cost implications for investment shells.

# 3.3.2. Modulus of rupture at room temperature

The results of MOR tests on green unfired samples are listed in Table 5. Polymer modified moulds exhibited higher green strength than that of the fiber modified mould, giving 7.8 and 4.7 MPa, respectively. After normalising for the shell thickness, the same effect was found in the AFL measurement, giving a fracture load of 16.6N for polymer modified mould and 13.8N for fiber modified mould. The unfired stress/strain curves are shown in Fig. 6. Suprisingly, no toughening effect was observed in the fiber modified system as would be expected with a composite system of this nature.

Microstructural examination of the fracture surface of the green fiber modified mould showed that most fibres were pulled directly out from the ceramic network, see Fig. 7a. The fibres also appear to be intact, with straight chopped ends and no "necking" as supplied. This suggests that there was little resistance and little force required to pull the fibres from the matrix, which explains the relatively low green strength recorded for these samples. The low resistance is most likely a result of the very smooth nature of the nylon fibre surface, illustrated earlier in Fig. 4 and again in Fig. 7b where the fibre cavity after pull out has very smooth walls. Future work will concentrate upon producing fibres with much rougher surfaces. This will increase the friction between fibre and matrix, increase the energy required for fibre pull out and ultimately increase the green strength of the ceramic shell. The results of AFL and MOR tests on fired samples are shown in Table 5. After firing, both polymer and fiber are totally burn out.

(a) 8 6 Sample-1 Stress (MPa) Sample-2 Sample-3 4 Sample-4 × Sample-5 2 Polymer Modified Mould -green 0 0.5 1.5 2 0 1 Strain (%) (b) 8 Sample-1 Sample-2 Sample-3 Sample-4 × 6 Sample-5 Stress (MPa) 4 2 Fibre Modified Mould -green 0 2 0.5 0 1 1.5 Strain (%)

Fig. 6. Showing the typical unfired stress/strain behaviour of (a) polymer modified mould and (b) fiber modified mould.

The flat section strength of fired samples is nearly identical, giving 4.7 MPa and 4.8 MPa respectively. This suggests that the higher green strength of polymer modified mould is a direct result of the polymer content alone. The fired stress–strain are illustrated in Fig. 8. A similar fracture behavior was found in these two systems. Therefore, due to thicker shell build, a higher AFL in the fiber modified system was found compared to that of the polymer modified mould, being 12.6 and 10.0 N, respectively. This gives the fiber modified mould a 26% increase in load bearing capacity and potentially

Table 5

Comparison of the 3-point bending test results of the two shell system

Mould sample	Status	No. of samples	Shell thickness (mm)	Modulus of rupture (MPa)	Adjust fracture load (N)
Polymer modified mould	Green	12	4.6	7.8	16.6
	Fired	12	4.6	4.8	10.0
Fiber modified mould	Green	10	5.3	4.7	13.8
	Fired	10	5.2	4.7	12.6





Fig. 7. Secondary electron microscopy image showing the green fracture surface of fibre modified ceramic shell (a) fibres pulling out from the surface; (b) the fibre cavity after pull out.

a better resistant to the mould wall bulge and related defects of the casting components.

Although higher strength and load bearing capacity (AFL) in the green polymer modified system indicates a higher ability to resist cracking during dewax. In practice, however, moulds produced with fibre additions are less susceptible to autoclave cracking.<sup>7</sup> As most cracking of mould during dewax and casting is at the sharp radii and corners, further comparison of strength and load bearing capacity using edge strength testing was carried out.

# 3.3.3. Wedge test

The strength and load bearing capacity of the moulds at sharp edges was accessed using the wedge test. A good correlation between edge and MOR strength was found in both systems. Wedge test results are shown in



Fig. 8. The typical stress/strain curve of the ceramic moulds after firng (a) polymer modified mould and (b) fiber modified mould.

Table 6. It can be seen that the strength of the polymer modified system is still slightly higher when the moulds are green and fired. However, due to the significant increase in the wall thickness at the edge, the fibremodified system exhibits a much greater load bearing capacity (AFL) in both the green and the fired states, showing a 65% increase in the green state and a 85% increase after firing. This explains why much less cracking is observed in practice with the fiber system as most mould failure occurs along the sharp edge. The wedge test examines the weakest part of a mould and is therefore is a better quality control assessment than flat bar MOR. Results show that introducing a fiber modified shell system could lead to an equivalent bulk ceramic thickness shell at flat section produced using fewer coats. The moulds would still have thicker wall thickness at the sharp corners, comparing to the current polymer modified system. This could provide sufficient mechanical properties at edges to reduce mould failure whilst significant reducing production time and material cost.

Table 6					
Comparisons	of the	wedge	test	results	

Mould sample	Status	No. of samples	Shell thickness (mm)	Modulus of rupture (MPa)	Adjust fracture load (N)
Polymer modified mould	Green	10	2.5	1.6	1.7
	Fired	12	2.4	1.4	1.4
Fiber modified mould	Green	10	3.5	1.3	2.8
	Fired	10	3.5	1.2	2.6

#### Table 7

Ceramic permeability comparisons

Mould Sample	No. of samples	Temperature (°C)	Test duration (min)	Permeability $(m^2 \times 10^{-7})$	Standard Deviation $\sigma$ (m <sup>2</sup> ×10 <sup>-7</sup> )
Polymer modified mould	3	800	5	1.612	0.183
·	3	800	10	1.601	0.152
	3	800	15	1.625	0.126
Fiber modified mould	3	800	5	5.792	0.239
	3	800	10	5.458	0.322
	3	800	15	5.435	0.334

### 3.3.4. Permeability

To ensure the quality of casting manufactured by the investment casting process, the mould has to be sufficiently permeable to obtain complete mould fill during casting. A comparison of the permeability of the fired shell samples at 800 °C can be seen in Table 7 and represented graphically in Fig. 9. Extending the dwell time from 5 to 15 min gave no significant effect on permeability, which indicated no further sintering occurs during the test. The addition of nylon fibres dramatically increases the fired permeability by a factor of 3 at casting temperatures, mainly due to increased porosity



Fig. 9. Comparison of permeability at 800  $^\circ\mathrm{C}$  for the fired shell samples.

networks left after the fiber burn out. This is of great benefit to the casting process, allowing increased transport of air displaced from the mould cavity.

# 4. Conclusions

Properties of an investment casting mould prepared using organic fibre additions were investigated in this work. Nylon fibres, with a diameter of approximately 20  $\mu$ m and a length of 1 mm, were added into a water based silica binder to replace the costly liquid polymer addition. The use of fibres increases the shell thickness by a factor of 13% on flat section and 40% at edges over that of a comparable system using a polymer modified binder. This equates to the ability to reduce the number of coats applied to the wax, which has significant production time and material cost implications for foundries.

Comparison of green strengths measured in 3-point bend mode suggests that the fibre addition produces a shell system with a slightly lower load bearing capacity on flat section, but a 65% increase in load bearing capacity was found at edges measured by the wedge test. After firing, when both polymer and the fiber are totally burn out, higher load bearing capacity was found in fiber modified system on both flat section and edge samples, giving 26 and 85% increase over the polymer system respectively.

The results indicate that fibre can be used to produce an equivalent ceramic thickness with fewer coats. This shell would still be thicker at the sharp corners providing enhanced mechanical strength at the point of prevalent failure. Thus, an improved performance of ceramic mould can be achieved, with a significant reduction in production time and material cost for investment casting industry.

Another significant advantage of the fibre additions appears to be a greatly increased permeability after firing. The addition of fibre increases the permeability at casting temperatures by a factor of 3. The permeability is unaffected by time at temperature and remains consistent throughout the test. This is of great benefit to the casting process, allowing increased transport of air displaced from the mould cavity and reducing defects associated with air entrapment.

The unique properties of the fibre modified system make it a promising alternative for the standard investment casting foundry.

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