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The influence of autoclave steam on polymer and organic fibre modified ceramic shells

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Abstract

The removal of wax from an unfired ceramic shell system without cracking or dimensional alterations is a key stage within the investment casting process. The effect of autoclave steam on polymer and fibre modified investment casting mould behaviour has been investigated. The polymer modified system exhibited a higher mechanical strength in the green dry state, but that strength significantly reduced when subjected to a simulated autoclave "wet" condition, giving a 38% reduction in a flat bar section and a 45% reduction in an edge test. It is suggested that this is related to the softening of the latex particles when they are in contact with steam. In comparison the fibre modified system showed a much lower reduction in strength when subjected to "wet" conditions. Calculating the adjusted fracture load (AFL) bearing capacity for the extra shell thickness of the fibre system showed that the fibre system outperforms the polymer system when the samples were tested "wet", showing a 33% increase in a flat bar section and an increase of over 150% in the more vulnerable edge region. The results suggest that the effect of moisture must be taken into account when studying the shell behaviour under autoclave conditions.

Keywords: Investment casting; Mould; Fibre; De-wax; Steam

1. Introduction

Investment casting, which is also called precision casting, is an effective way to obtain dimensionally accurate metal components. Compared to other modern techniques for "precision" casting fluid metals, for instance gravity die casting, pressure die casting, plaster mould casting and shell sand mould casting, investment casting is the most flexible with respect to attainable intricacy, precision and variety of alloys which may be cast within the inherent size limitations.[1] The manufacture of an investment casting requires an expendable pattern, which is coated with multi-component slurries, or 'investments', to form a ceramic mould.[2,3] The pattern is dipped into a ceramic facecoat slurry, sprinkled with a coarse grained refractory 'stucco' and dried. The dipping and stucco coating process is repeated many times to produce a graded mould. Flexibility exists in changing the composition of each layer. The particle size of the stucco is increased as more coats are added to maintain maximum mould permeability and to provide bulk to the mould. The pattern is then removed, leaving a hollow mould with an extremely smooth internal surface. Moulds are fired and cast with molten metal. After cooling, the ceramic is removed by mechanical or chemical methods to obtain the metal parts. In recent years, this process has increasingly been used to produce components for the aerospace industry and it has been particularly successful for the production of single crystal turbine blades.[2,4–6]

Most investment foundries use wax as the pattern material.[7–10] Different methods can be used to remove the wax pattern such as steam autoclaving[11,12] and flash-firing[13] to leave the hollow shell. The steam de-wax process involves exposing wax and shell to steam at a high pressure and a relatively high temperature within a sealed vessel. Heat is transferred rapidly to the wax surface causing the wax to melt and flow from the cavity. However, both wax and ceramic will expand during heating and as a consequence the weak unfired ceramic shell is prone to cracking during this stage of the process. Polymer is normally added to the ceramic slurry to increase the green strength of the ceramic shell.[14,15] However, liquid polymer additions are relatively expensive and previous work has shown that the

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green strength of a polymer modified shell is reduced significantly when placed in a steam bath for a relatively short period of time.[16] This has implications with regard to crack resistance during the de-wax cycle. One recently patented method of using organic fibre to replace liquid polymer additions has been intensively investigated by Yuan and Jones.[17,18] The fibres, known as WexPerm®, are organic and insoluble in water. It was found that fibre addition greatly increased the shell thickness especially on edges. Furthermore, higher permeability was developed in fibre modified system once the shell is fired due to the cavity left by the volatilised fibre. This allows increased transport of air displaced from the mould cavity and reduces casting defects associated with air entrapment.[17,19]

In this paper, the reported experimentation was designed to determine and compare the effect of autoclave steam on the properties of a polymer modified ceramic shell system and a fibre modified shell system. The polymer used here was latex and the fibre was nylon. The green strength was measured under traditional dry conditions and also under simulated autoclave condition. Fired strength and Young's modulus of these two systems was also investigated in this work.

2. Experimental procedures

2.1. Organic fibre specification

The nylon fibres used in this study are typically 1 mm in length, with an average diameter of 20 μm, as shown as in Fig. 1, giving a aspect ratio of 50:1. The selected loading rate was 20 g/l of binder liquids.

2.2. Ceramic shell specifications

The ceramic shell applied in this study was designed to be representative of a standard shell used for aluminium alloy casting. Details of slurry composition and mould build are given in Tables 1 and 2.

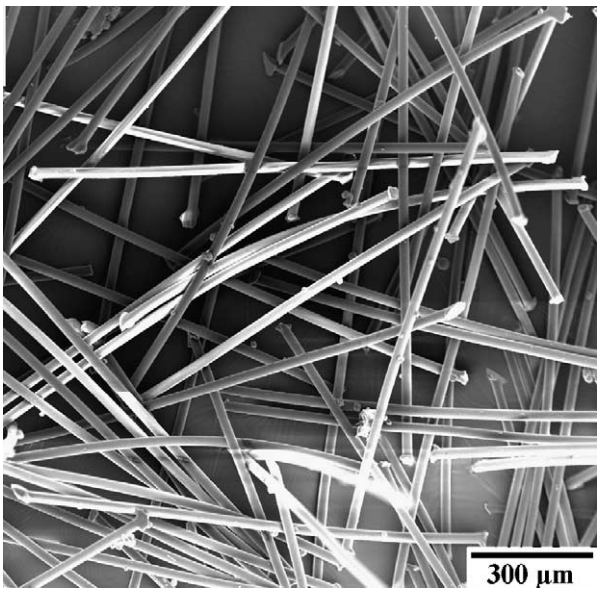

Fig. 1. Secondary electron microscopy image showing 1 mm nylon fibre additions.

The primary slurry, which would ultimately be in contact with molten alloy, consisted of a colloidal silica binder (Remet, LP-BV), zircon filler (Johnson Matthey, −200 mesh Zircosil), liquid polymer (Remet, Adbond ADII), wetting agent (Remet, Victawet 12) and anti-foam (Remet, Burst RSD-10). The same primary slurry was used for both shell system.

The polymer modified backup slurry contained 8 wt.% polymer of total liquid, while the fibre modified backup slurry contained nylon fibre (Wex Chemicals Ltd., WexPerm™) at a ratio of 20 g/l (1.8 vol.%) 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

Table 1
Slurry compositions for ceramic mould samples

Slurry	Materials	Composition
Primary	LP-BV silica sol[a]	25 wt.% silica of colloidal sol
	Adbond ADII polymer[a]	6 wt.% of total liquids
	Victawet 12 wetting agent[a]	0.3 wt.% of total liquids
	Burst RSD-10 antifoam[a]	0.5 wt.% of total liquids
	−200 mesh Zircosil[b]	77 wt.% refractory loading
Secondary (polymer modified)	LP-BV silica sol[a]	25 wt.% silica of colloidal sol
	Adbond BV polymer[a]	8 wt.% of total liquids
	−200 mesh Alumino-silica[a]	57 wt.% refractory loading
Secondary (Fibre modified)	Silica sol 1030[c]	25 wt.% silica colloidal sol
	WexPerm™ fiber[c]	20 g fibre per litre of liquid (1.8 vol.%)
	−200 mesh Alumino-silica[a]	57 wt.% refractory loading

[a] Remet, UK.
[b] Johnson Matthey Ceramics Ltd.
[c] Wex Chemical Ltd.

Table 2
Shell Build for polymer modified and fibre 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

Key: M: Molochite^{TM} (alumino-silicate); 30/80 = 30/80 mesh stucco; 50/80 = 50/80 mesh stucco.

rainfall sanding method. The coarse grains pass through a suspended agitated mesh (1 m height) fall and 'showered' down onto the wax assembly. The assembly was rotated 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 21 °C, 50% relative humidity and 0.4 ms^{-1} 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 3 ms^{-1} air speed for 90 min. Finally a seal coat of secondary slurry was applied and dried at a temperature of 21 °C, 50% relative humidity and 3 ms^{-1} air speed for 24 h. The wax inside the ceramic mould was then removed by steam autoclaving at 8 bar maximum pressure for 4 min, followed by a controlled de-pressurisation cycle at 1 bar/min using a Quicklock Boilerclave^{TM} (Leeds and Bradford Boiler Company Ltd., UK).

For flat bar mechanical testing, the samples were prepared upon a wax pattern with dimensions of 200 mm × 25 mm × 10 mm thickness. After de-wax, the moulds were cut into rectangular test bars. For edge testing, the test pieces were taken from moulds produced using a specially designed wax pattern, as shown as in Fig. 2, which produces symmetric edge sections. The length of the edge test sample was approximately 20 mm and the width of the sample 10 mm. To measure properties of shell after firing, one-third of samples were fired at 1000 °C for 60 min.

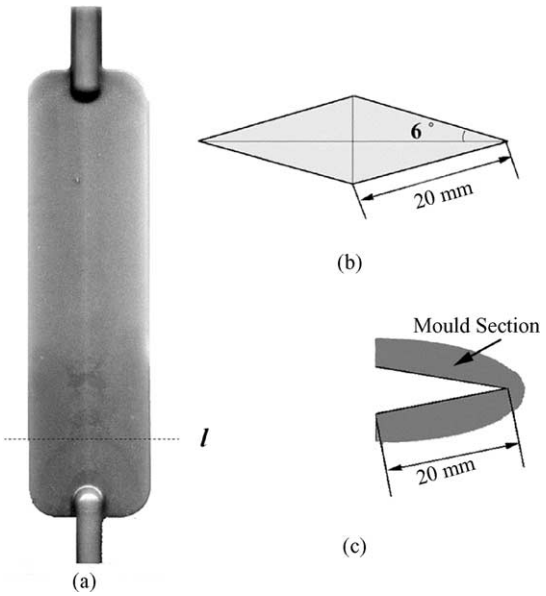

Fig. 2. Showing (a) the wax pattern for edge test; (b) the configuration of the wax pattern section *l*; and (c) the configuration of the ceramic edge test piece.

2.3. Flat bar property measurement

Green strength measurements were carried out under "wet" and "dry" conditions. For the "wet" condition, samples were put above a steam bath for 15 min to simulate the autoclave de-wax procedure and tested immediately after removal. For the "dry" condition, samples were dried for 24 h at room temperature prior to the tests, which is the normal test procedure most foundries adopt to determine green shell strength. Samples were loaded in a three point bending test geometry on an Instron 8500 tensile testing machine. The span length, L, was 50 mm. A load application rate of 1 mm/min was used. The failure strength, σ_{Max} was calculated using

$$\sigma_{Max} = \frac{3P_{Max}L}{2WH^2} \quad (1)$$

where P_{Max} is the fracture load, W and H are the width and thickness of sample fracture area, respectively. The load/deflection curve was converted into a stress/strain curve using

$$\sigma = \frac{3PL}{2WH^2} \quad (2)$$

$$\varepsilon = \frac{6H\delta}{L^2} \quad (3)$$

where σ is the stress, ε is the strain, δ is the deflection and P is the corresponding load. The Young's modulus E, which represents the stiffness of a material, is estimated from the stress/strain curve using linear regression of stress/strain plots.

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[20]

$$AFL_B = f_B\sigma_{Max}H^2 \quad (4)$$

where f_B is a constant equal to 0.10.

2.4. Edge test

The edge, or wedge test[21] is specifically designed as a quality control procedure to determine the actual strength

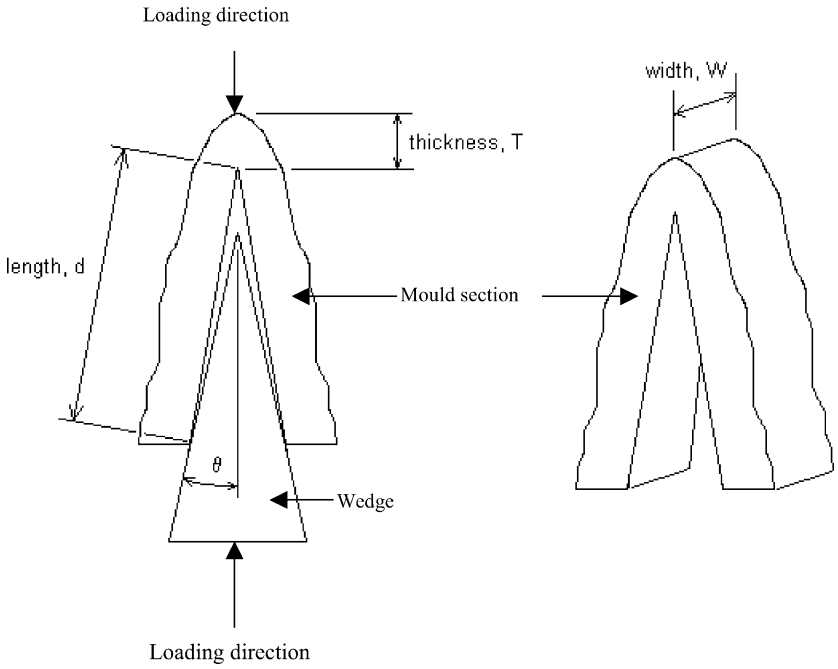

Fig. 3. The schematic loading configuration for the wedge test.

of the ceramic shell at the most vulnerable edges where strength and shell build are vital to prevent cracking during de-waxing and casting. Edge testing was also conducted under "wet" and "dry" conditions. Instead of testing a plane mould surface, a specially designed 29° wedge was forced into a V shape 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[22]

$$\sigma_{\text{Wedge}} = 12.2\frac{\sin\theta\cos\theta Fd}{WT^2} \quad (5)$$

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)

$$AFL_W = f_W\sigma_{\text{Wedge}}T^2 \quad (6)$$

where f_W is a constant equal to 0.17.

3. Results and discussion

3.1. Shell build comparisons

Comparisons of the ceramic shell thickness achieved for polymer modified and fibre modified shell systems can be seen in Table 3 and Fig. 4. In both cases, the shell build on

Table 3
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

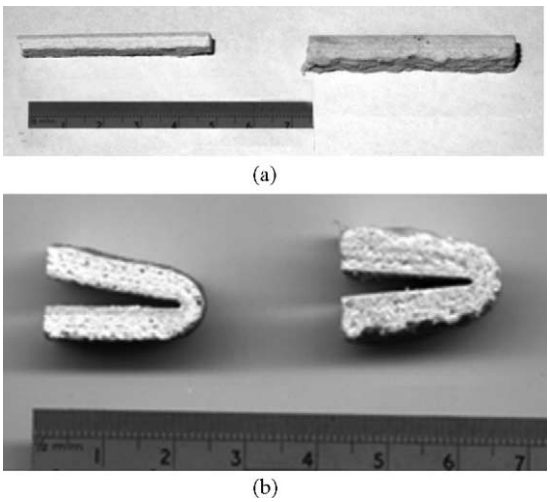

Fig. 4. Image highlighting the shell thickness increase produced with organic fibre modified slurries (a) flat bar section of polymer modified shell (left) and fiber modified shell (right), (b) edge test pieces of polymer modified shell (left) and fiber modified shell (right).

the edges is much less than that obtained on flat bar samples. This is related to the extent of draining of the slurry at the edge and the difficulty of stucco adherence onto the wet slurry coat around the angled morphology. The use of fibres increases the flat shell thickness by 0.66 mm (14%) and the edge build thickness by 0.83 mm (30%) over a primary + four coat shell build.

3.2. Mechanical properties of flat bars

Comparison of the strength and load capacity achieved under dry conditions for the green shell samples is shown in Table 4. Fibre reinforced ceramic has a lower green strength than the polymer modified system, being 4.7 MPa as compared to 7.8 MPa. The same trend was also found in the adjusted fracture load bearing capacity (AFL) measurement, giving a fracture load of 12.7 N for the fibre modified mould and 16.6 N for the polymer modified mould.

The high green strength of polymer modified shell is a direct result of the polymer content, which is reflected by the reduction in strength when the sample is fired at 1000 °C, as the polymer has been burnt out. The strengths of fired samples of the polymer and fibre systems are similar, being 4.7 and 4.8 MPa, respectively, as shown as in Table 4. The equivalent fired strength suggests that the extra volume of cavity left after fibre combustion is not acting as a significant defect which would reduce the strength of fired shell. However, due to thicker shell build, a higher AFL in the fibre modified system was found compared to that of the polymer modified mould, being 12.6 and 10.0 N, respectively. This gives the fibre modified mould a 26% increase in load bearing capacity and potentially better resistance to mould wall bulge and related defects within the cast components.

Although the polymer modified system exhibits a higher strength in the green dry stage, in practice, moulds produced with fibre additions are less susceptible to autoclave cracking.[16,19] It has also been suggested that the dry green strength is not an accurate measure of the shell crack resistant during hydrothermal de-wax procedures.[16] The atmosphere (temperature and moisture) has a great influence on shell properties and has to be taken into account. Therefore, in this work a comparison of strength and load bearing capacity under "wet" condition was also carried out and the results are given in Table 4. It was found the strength of polymer modified shell dropped significantly from 7.8 to 4.8 MPa when it was tested "wet". This could be related to the softening of the latex particles when in contact with steam which reduces the adherence between the latex and the surrounding colloidal network. The colloidal network itself also reabsorbs moisture to a certain extent and will weaken. The Young's modulus derived from stress/strain curve also suggested that samples under wet condition are much less rigid than that under dry conditions, giving 5.94 GPa under dry condition and 3.26 GPa under wet condition, as shown in Table 5.

In comparison to the polymer modified system, the fibre modified shell shows much less reduction in strength and Young's modulus when subjected to simulated autoclave conditions, as listed in Table 4. The slight reduction in strength may again be due to the effect of moisture upon the colloidal gel network where reabsorbing of moisture softens the colloidal network bonds.

The above results show that both polymer and fibre modified shells exhibit a similar MOR green strength under the simulated autoclave condition. The polymer can no longer act as a strengthening material when subjected to the high temperature and the presence of moisture. The adjusted fracture load bearing capacity (AFL) of the fibre system is higher than the polymer when the samples are wet. This explains why in foundry observation, fibre modified shells are stronger and less susceptible to cracking in the autoclave.

3.3. Edge strength

Most autoclave shell cracking and damage occurs on the edges of components, such as trailing edges of turbine blades and sharp corners, where reduced shell build and high stress results in ceramic failure.[21,22] In order to fully understand the mechanical behaviour of the ceramic shell, it is necessary to further compare the two systems in this most vulnerable region.

The comparison of the edge strength results are listed in Table 6. It can be seen that the strength of the polymer modified system was still slightly higher when the moulds were green and dry. However, due to the significant increase in the wall thickness at the edge, the fibre-modified system exhibited a much greater load bearing capacity (AFL), showing a 65% increase over the polymer shell in the green dry state. The green strength of the polymer system also

Table 4
Comparisons of the flat bar results (wet and dry)

Mould sample	Status	Strength (MPa)	AFL ($f = 0.10$ N)
Polymer modified mould	Green, dry	7.8 ± 0.7	16.6 ± 1.9
	Fired	4.8 ± 0.3	10.0 ± 1.2
	Green, wet	4.8 ± 0.3	9.0 ± 2.2
Fiber modified mould	Green, dry	4.8 ± 0.5	12.7 ± 1.1
	Fired	4.7 ± 0.7	12.6 ± 1.5
	Green, wet	4.7 ± 0.6	12.0 ± 1.5

Table 5
Comparison of Young's modulus of flat bar samples

Mould sample	Status	No. of samples	Young's modulus (GPa)
Polymer modified mould	Green, dry	12	5.94 ± 1.10
	Green, wet	10	3.26 ± 0.57
Fiber modified mould	Green, dry	10	3.67 ± 0.49
	Green, wet	10	3.46 ± 0.76

Table 6
Comparisons of the wedge test results

Mould sample	Status	Strength (MPa)	AFL ($f = 0.17$ N)
Polymer modified mould	Green, dry	1.58 ± 0.22	1.70 ± 0.30
	Fired	1.44 ± 0.26	1.41 ± 0.34
	Green, wet	1.07 ± 0.32	1.00 ± 0.16
Fiber modified mould	Green, dry	1.26 ± 0.12	2.98 ± 0.65
	Fired	1.15 ± 0.19	2.56 ± 0.57
	Green, wet	1.10 ± 0.27	2.52 ± 0.62

decreases significantly (32%) when the samples are wet. The fibre system showed much less reduction (13%) when the samples were wet and the load bearing capacity was much higher (152%) than the polymer system.

Again, these results demonstrate why much less cracking was observed after the autoclave procedure with fibre modified moulds. In practice most mould failure occurs along sharp edges. The wedge test examines the weakest part of a mould and, therefore, is a better quality control assessment of such failure than flat bar strength. Results show that introducing a fibre modification could allow production of an equivalent bulk ceramic thickness shell (flat section) using fewer applied coats. The moulds would still have greater wall thickness on the sharp corners, compared to the current polymer modified system. This could provide sufficient mechanical properties at edges to reduce mould failure whilst significantly reducing production time and material costs.

4. Summary and conclusions

The use of fibres increased the flat shell thickness by 0.66 mm (14%) and the edge build thickness by 0.83 mm (30%) over a primary + four coat shell build. The polymer modified ceramic exhibited a higher dry, green strength than the fibre reinforced system and equivalent fired strength.

It was found that the strength of the polymer modified shell dropped significantly when it subjected to simulated autoclave conditions, while the fibre modified system showed less reduction in strength when it was tested in the same conditions. Adjusting for variance in shell build thickness, the adjusted fracture load bearing capacity (AFL) of the fibre system was higher than the polymer when the samples were wet, showing a 33% increase for flat test specimens and over 150% in edge test specimens. These results demonstrate why moulds produced with fibre additions in foundries are much less susceptible to autoclave cracking.

It can be concluded that the effect of moisture must be taken into account when studying shell behaviour under autoclave conditions. Fibres can be used to produce shells which exhibit greater retention of green strength in the autoclave, increased shell build per layer and improved edge strength. This leads to the possibility of using fewer coats for equivalent ceramic thickness and mechanical performance with a significant decrease in production costs.

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