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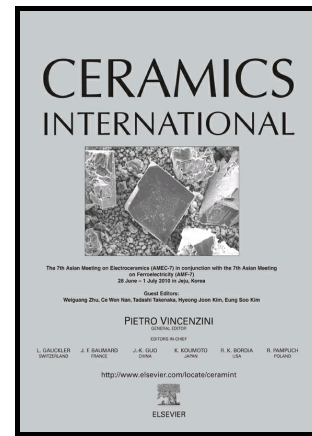
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Abstract

The influence of de-waxing method and post de-wax treatment on the flexural strength of ceramic shells has been evaluated. De-waxing by autoclave generates the highest strength when dry samples are tested, significantly stronger than when de-waxed by refrigerating. The autoclave-dewaxed shells were found not to be fully de-waxed but to have a coating of wax and some wax penetration into the face coat. This and the curing of the sol by the autoclave process were shown to be responsible for the higher strength.

Treating the shells after initial de-waxing to simulate the conditions in the autoclave by either boiling in water, steaming above water or autoclaving at 180 °C and 0.8 MPa caused changes in the reported flexural strength. For samples initially de-waxed by autoclave the strength was reduced, boiling caused the greatest strength reduction and re-autoclaving gave the least change. Testing wet always gave lower strengths compared to dry. However, initial dry strength was never regained on further drying. The samples prepared by refrigeration lost strength when boiled or steamed but gained strength on autoclave treatment. The strength built to be 57% of the samples de-waxed by autoclave in the dry state. This occurred because the sol cured; however, the shells being essentially free from wax never gained the strength of the samples de-waxed initially by autoclave.

Keywords: Mechanical characterisation, Ceramics, Heat treatment, Investment casting, Silica sol aging

Introduction

Investment casting is used to manufacture complex metal parts and had an estimated world part value of US\$ 11.6 Billion in 2012 [1]. It is favoured where the surface finish and tolerance requirements of the finished component are high. In some applications it allows unique metallurgical properties to be developed such as in the casting of single crystal components for the aerospace industry. The process normally starts with a wax pattern of the component to be cast [2, 3] which is then coated in refractory ceramic by dipping into a slurry comprising a sol and filler combination. This wet coating is sprinkled (stuccoed) with refractory grain before drying. The coating process is repeated until a layered shell structure of the desired thickness is attained. The first or primary coat is often made of a different composition to the further backup coats. The wax pattern is then removed from the shell in a process known as de-waxing and the shell fired to give strength and structure, and remove any remnant wax. The final strength of the shell is only developed during the firing stage where sintering of ceramic particles occur. Strength of the shell is different in each stage of the process and shell failures can occur at all stages but an area of particular concern is the de-waxing stage when the wax is removed from the 'green' shell. The number of failures at this stage depends on the wax composition, the shell make up and the complexity of the part. When a new part, shell system or wax is introduced to the process it would be advantageous to be able to predict the propensity for cracks to form during the de-waxing process.

In order to model the process the most appropriate data should be available to the modeller. One key variable is shell strength which because of the shells inhomogeneous and developing structure is almost certainly not constant under the conditions of the de-wax process. In the raw green state (before de-wax) shell strength comes from a bond formed by the gelation/coagulation of the sol contained in the slurry during drying. It is this rather weak structure which has to take the stresses of removing the wax and is partly why cracking of the shell can occur during de-wax. It is not the whole story, the process is

complicated by the wax having a higher thermal expansion coefficient than the green shell that now surrounds it but fortuitously this is countered by the wax's lower thermal conductivity. Without rapid heating these features would combine to cause the shell to frequently fail in tension. Two dewaxing methods are typically used to remove the wax from the shell: autoclaving [4, 5] and flash firing [5]. Steam autoclaving is the most common method, in this process steam is injected at high pressure into a sealed chamber to raise the temperature and pressure to typically around 180°C and 0.8 MPa respectively in a matter of seconds. Heat from the steam travels through the shell and melts the wax. The surface melting of the wax relieves the pressure on the shell either by drainage from the mould or absorption into the porous shell structure, however, if the expansion stresses imposed on the shell overcome the shell's mechanical strength in the green state failure will occur. The wax removal process used often leads to products containing some remnant of the wax after de-waxing. Jones *et al.* [6] report different test methods where autoclave de-waxed test bars were either soaked in boiling water or returned to the autoclave for a further cycle (putting the shell bars twice in the autoclave) and testing them immediately. It was found that the strength of the shell was significantly reduced to about 50% of the post autoclave dry results. In a further paper, Jones [7] also treated the autoclave de-waxed ceramic shell with steam by suspending the test sample over boiling water and testing the mechanical strength immediately after steaming. With this method variable results were reported depending on the shell type but typically strength loss was in the range 10-50% of the post autoclave dry strength. In both papers the shells were prepared by using autoclave de-waxing, thus the strength of the ceramic shell will have been affected by the extent to which the wax had penetrated into the shell and the damage induced by the de-waxing process. Mechanical testing of the dried autoclave dewaxed specimens would give the normal handling strength of the shell before firing. This represents the most typical condition the foundry sees after dewax but before firing.

In addition to the conventional processes described above the shell may be released from the wax by cooling. The wax shrinks more than the ceramic and therefore separates. It results in shell samples that are wax free and not treated by heat above that required for moisture removal. Comparing dewaxing by cooling and by autoclave would allow the influence of these phenomena to be examined. Branscomb [8] determined the flexural strength of ceramic shells without a prime coat at various temperatures and following various preparation conditions. The samples were prepared by carefully removing the shell from the wax without heating. Branscomb prepared the shell with different compositions of slurry and the flexural strength was determined at different temperatures and conditions – 21°C dry, 93°C dry, 232°C dry, 21°C wet and 93°C wet. However, for the wet test it is unclear if the shells were dipped in water and tested or the shell was tested while submerged in water. Here again a general loss of strength was reported when the materials were wet compared to dry, with values ranging from 11 to 54% lower depending on condition.

The aim of this study was to understand the development of shell strength in the green and autoclaved state. These results can be used to represent mechanical strength of shell when modelling the autoclave de-waxing processes commonly used in the investment casting industry.

Experimental Procedures

Wax bars measuring 200 x 30 x 5 mm were formed by injection moulding a filled pattern wax. The wax patterns were cleaned with wax cleaner solution and dried before being dipped into a primary ceramic slurry, the composition of which is given in Table 1. The primary slurry coat was stuccoed by hand with a zircon grain the specification of which is given in Table 3. A further 6 coats were applied with a secondary slurry composition given in Table 2 and hand sprinkled with a molochite stucco described in Table 3. Between each slurry coat the bars were dried at 50% RH and 21 °C. A final seal coat of the secondary slurry was applied before a final dry under the same conditions. The primary slurry had about four times higher viscosity than the secondary slurry. The stucco compositions are given in Table 3 along with the dipping and stuccoing sequence.

Table 1- Standard steel primary slurry composition

Initial formulation	Mass(%wt)
Filler (200 mesh)	79.83
Silica Binder	17.00
Polymer	1.20
Wetting Agent	0.06
Antifoam	0.10
Deionised water	1.81

Table 2- Standard steel secondary slurry composition

Filler (200 mesh)	43.05 fused silica (200mesh)&14.37 Molochite (200 mesh)
Silica Binder	32.83
Polymer	3.34
Wetting Agent	-
Antifoam	0.22
Deionised water	6.19

Table 3- Shell coating specification for steel casting

Slurry	Stucco	Dip time, s	Drain time, s	Air speed, ms ⁻¹	Dry time, h
Primary	Zircon sand	30	60	0.4	24
Secondary 2-3	30/80 Molochite	30	60	3	1.5
Secondary 4-7	16/30 Molochite	30	60	3	1.5
Seal	-	30	60	3	24

Bars for mechanical testing measuring 50 x 20 mm x the shell thickness were prepared by either de-waxing the 200 x 30 x 5 mm shelled wax by conventional autoclave treatment with a dwell at 0.8 MPa and 180°C for at least 3 minutes, followed by a controlled depressurisation cycle at 0.33 kPa·s⁻¹ or by first cutting the 200 x 30 x 5 mm shelled wax to the desired shape followed by chilling overnight (12 hours) at around 5°C to release the wax from the shell. Figure 1 illustrates preparation of test bars by the chilling method. All cutting was undertaken by diamond saw.



Figure 1- Fridge de-waxing: the shelled flat bar wax was cut after drying to the required test bar dimensions and then separated by cooling in a fridge. The picture on the right shows how clean the ceramic bars are after the de-wax process.

The bars were stored under atmospheric conditions until mechanical strength evaluation or further treatment was undertaken. A number of these samples were re-entered into the autoclave and heat treated with the same cycle described above giving a total of four different bar types for subsequent mechanical testing. These test bars were treated in one of three ways before testing. They were either tested as stored, boiled in water for 20 minutes or steamed over boiling water for the same duration (with each of the last two options the material could be tested wet directly after treatment or dried before testing). Additionally the bars could be evaluated with prime coat in tension or seal coat in tension. This gives 48 permutations of the experiment, however not all were evaluated and the 16 selected permutations are given in Table 4.

Table 4-Summary of experimental methods for sample preparation and mechanical testing

Initial dewax method	Autoclave for second time	Post dewax treatment	Tested dry or wet	Tested primary up or down	Method notation
Autoclave	No	Dry		Up	1(a)
				Down	1(b)
		Wet		Up	
				Down	
		Steam	Dry	Up	
				Down	
			Wet	Up	
				Down	
		Boil	Dry	Up	
				Down	
	Wet	Up	3(a)		

	Yes	Dry		Down	3(b)		
				Up	9		
		Wet		Down			
				Up	5		
		Steam	Dry		Down		
					Up	11	
			Wet		Down		
					Up	10(a)	
			Dry		Down	10(b)	
					Up		
		Boil	Wet		Down		
					Up		
		Fridge	No	Dry		Up	2(a)
						Down	2(b)
Wet				Up			
				Down			
Steam	Dry			Up			
				Down			
	Wet			Up			
				Down			
Boil	Dry			Up	7		
				Down			
	Wet			Up	4(a)		
				Down	4(b)		
Yes	Yes			Dry		Up	8
						Down	
		Wet		Up	6		
				Down			
		Steam	Dry		Up		
					Down		
			Wet		Up		
					Down		
		Boil	Dry		Up		
					Down		
			Wet		Up		
					Down		

For each tested permutation a minimum of 10 bars were tested. It is important to note that the mechanical strength of samples vary from batch to batch in these brittle and often rather weak ceramic structures. To compensate for this, the results listed in each table are from the same batch of samples. Comparison between tables and thus batch requires caution. The flexural strength of the shell was measured using a 3-point bend test geometry on Instron 4467 load frame with a 1 kN load cell and a loading speed of $1\text{mm}\cdot\text{min}^{-1}$ (0.0167mms^{-1}). The span was set at 50 mm. The failure strength of ceramic shell, σ_{max} was calculated using:

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

where P_{Max} is the fracture load, W and H are the width and thickness of sample fracture area respectively.

Results and Discussion

Part 1: Effect of different preparation method on shell mechanical strength

The flexural strength results are given in Tables 5, 6 and 7 for different batches of shell build and allow different interrelationships to be examined systematically. Some results are duplicated in Tables 5 and 7 and show that there is variability in the returned values batch to batch. For example, when samples produced by Method 1 are compared, the flexural strengths range from 5.99 to 6.26 MPa across the repeats. Thus only results within a particular data set and table will be compared. De-waxing by autoclave and testing with the primary coat in compression (method 1 (a)) gave flexural strengths approximately 2.7 MPa greater than samples prepared by the fridge de-wax method (method 2 (a)). The change in strength was the same when tested prime coat in tension (methods 1 (b) and 2(b)). This difference can be attributed to a combination of the penetration of wax into the shell and wax left on the surface of inner shell in the samples prepared in the pressurised autoclave environment and by any microstructural changes brought about by the thermal treatment.

Table 5- Shells samples prepared in different ways to observe the average strength

Method	Ceramic Shells			Average strength (MPa)	Standard deviation
	Dewax method	Post-dewax treatment	Mechanical testing method		
1(a)	Autoclave	-	Prime up (PU)	6.26	0.79
1(b)			Prime down (PD)	6.25	0.67
2(a)	Fridge	-	Prime up	3.56	0.22
2(b)			Prime down	3.57	0.35
3	Autoclave	boil	Wet (PD)	4.66(decreased after boiled)	0.47
4	Fridge	boil	Wet (PD)	0.91	0.11
5	Autoclave	autoclave	Wet (PD)	4.37	0.61

6	Fridge	autoclave	Wet (PD)	4.55(increased after autoclaved)	0.48
7	Fridge	boil	Dry (PD)	2.86	0.17
8	Fridge	autoclave	Dry (PD)	5.88	0.59

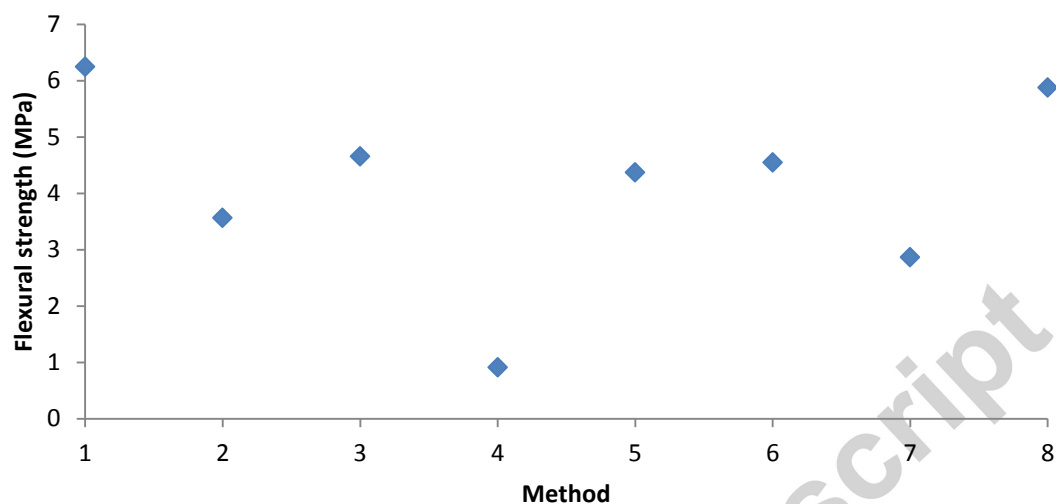


Figure 2- Graphical representation of the data presented in Table 5.

Table 6- Mechanical test to verify the effect of primary up or down on de-waxed boiled shell

Method	Ceramic Shells			Average strength (MPa)	Standard deviation (MPa)
	Dewax method	Post-dewax treatment	Mechanical testing method		
3(a)	Autoclave	Boil	Wet (PU)	3.99	0.59
3(b)			Wet (PD)	4.56	0.88
4(a)	Fridge	Boil	Wet (PU)	0.92	0.09
4(b)			Wet (PD)	0.85	0.10

Table 7- Mechanical test to observe the effect of steam on the shell strength

Method	Ceramic shells			Average strength (MPa)	Standard deviation (MPa)
	Dewax method	Post-dewax treatment	Mechanical testing method		
1	Autoclave	-	Primary down (PD)	5.99	0.42
3	Autoclave	Boil	Wet (PD)	4.28	0.24
5	Fridge	Autoclave	Wet (PD)	4.62	0.24
6	Autoclave	Autoclave	Wet (PD)	4.47	0.45
9	Autoclave	Autoclave	Dry (PD)	5.61	0.38
10(a)	Autoclave	Steam	Wet (PU)	4.48	0.59
10(b)			Wet (PD)	4.45	0.25
11	Autoclave	Steam	Dry (PD)	5.05	0.38

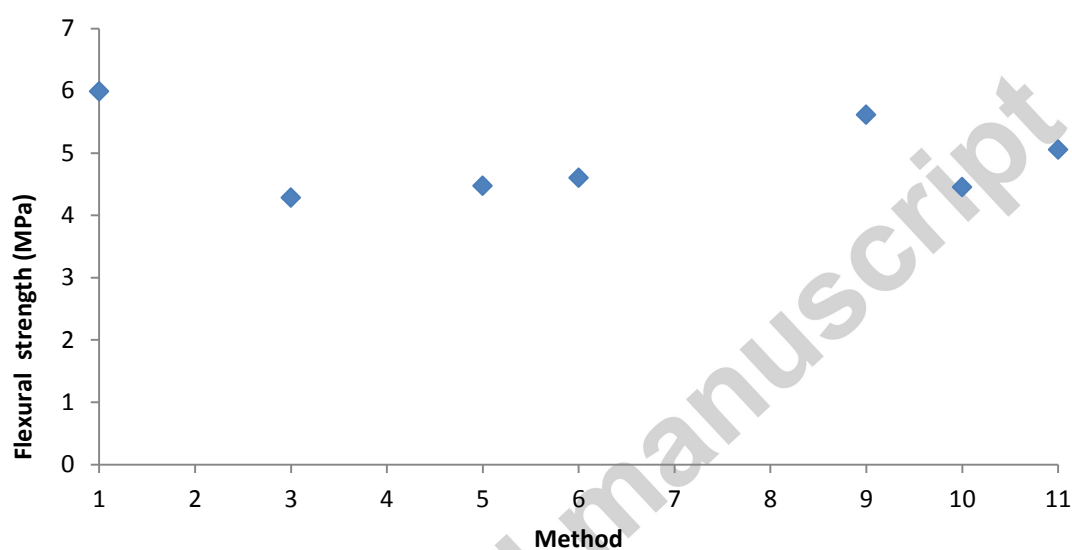


Figure 3- Graphical representation of the data presented in Table 7.

To investigate the influence of autoclave steam on flexural strength, the fridge de-waxed shells were heat treated in the autoclave at 180°C and 0.8 MPa for at least 3 minutes, followed by a controlled de-pressurisation cycle at 0.33 kPa·s⁻¹ (method 6 and 8). The strength increased by 0.98 MPa when tested wet and 2.31 MPa when the shell was dried after the autoclaving process. Two possible mechanisms for this strength increase are suggested:

- i. The shells could have been compressed by the autoclave pressure to form a more compact structure.
- ii. Autoclave treatment could have increased the coalescence of silica sol in the binder system.

The cause of the strength increase on the sol after autoclaving has been investigated further and those studies are discussed in Part 2.

Comparing method 1a with 1b and 2a with 2b (table 5) shows that testing the shells with primary coat in compression or tension did not lead any significant difference in strength (the error being significantly greater than the differences). However, there is a distinct difference when comparing the mechanical strength of shell when it is wet or dry either from autoclave steam or from treating in boiling water. Consistently the wet shells showed lower mechanical strengths than their dry counterparts. For example when fridge dewaxed samples are boiled in water the material has 68% less strength when wet compared to dry. With autoclave treatment the difference is less pronounced, for example fridge dewaxed samples autoclaved and tested wet gave a 25% reduction in strength compared to the equivalent dried material. This strength reduction can be attributed to the binder system being “softened” by the hot water or steam. Branscomb [8] observed similar behaviour where flexural strength dropped significantly when tested wet (11% drop) or hot and wet (23% drop).

When comparing shell de-waxed by refrigerating (method 2(b)) and with the same material boiled and then dried (method 7), the strength of shell reduced by approximately 0.7 MPa. This shows that the binder system is affected by the boiling water and drying does not restore the mechanical strength to the initial value recorded on wax removal. Shell strength reduces by approximately 2.7 MPa when the refrigerated shell is boiled and tested wet.

Method 3 showed that the flexural strength was reduced by 1.7 MPa (approximately 25%) after the boiling process if the sample remains wet when compared to method 1(b). This is to be expected as the water softens the silica sol used in the dried slurry and the result is consistent with data presented by Jones *et al.* [6].

Samples de-waxed in the refrigerator were significantly weaker than the materials prepared by autoclave de-waxing. When the refrigerator samples were boiled and tested wet (method 4) they further weakened by 2.65 MPa (approximately 75%). This is attributed to the damage induced by the boiling process and the weakly integrated network of the silica gel. It can be seen that the shell strength of the refrigerator de-wax samples (method 6) and method 3 used by Jones *et al.* [6] give similar strengths. This is because the initial autoclave de-waxed strength although being relatively high has significantly decreased after the soaking in the boiling process. The fridge de-waxed shells have lower mechanical strength prior to autoclave heat treatment but increase significantly after autoclaving due to either sol gel curing or compaction as discussed above.

Table 6 shows that autoclave de-waxed and boiled shell tested wet materials (method 3) gave slightly different results when the samples were tested primary up or primary down. Jones *et al.* [6] applied this method to examine shell strength and suggest it is important to choose which side of the shell is put into tension or compression. Primary down samples shows approximately 0.56 MPa higher strength compared to shells being tested with primary up. However, there is no significant difference when comparing the primary up or down of the fridge de-waxed and boiled wet shell (method 4). This is due to the wax

penetrating or sticking on to the primary coat in method 3 which restricted the hot water penetrating the binder system through the primary coat side during the 5 minutes of the boiling water soak. The autoclave de-waxed boiled wet shells (method 3) have higher standard deviation compared to fridge de-waxed boiled wet samples (method 4). It is postulated that the penetration of wax into shell and any excess wax sticking to the inner shell surface may vary in volume thus accounting for the greater variation in the shell strength. It is also possible that the wax penetration into the shell is responsible for the slightly higher strength recorded for method 1 compared to method 2.

In the work reported here it was observed that both shell treatment methods 5 and 10 (b) exhibited a 25% decrease in strength (Table 7) and both methods have similar mechanical strength. By comparison, fridge de-waxed and autoclaved wet shell (method 6) exhibited a slightly higher strength than de-waxed autoclaved wet shells (method 5). This is probably due to the shell weakening during the autoclave de-waxing process where the wax expands more than the shell mould. Another possibility is that the fridge de-waxed and then autoclave treated shells have incurred lower damage during cutting than the de-waxed shells because the wax bar supported the shell. If the samples are dried after being re-treated in the autoclave or steamed (methods 9 and 11 respectively) then there is partial recovery of the strength but not to that of the original autoclave samples tested dry (method 1(b)).

Part 2: Effect of Autoclaving treatment on the binder system

In the first part of the discussion two possibilities were proposed to account the increase in strength of shell after the autoclaving process: the collapsing of ceramic structure into a more compact form or the further coalescence of the silica sol by autoclaving.

To evaluate the shells dimensional changes ten samples of fridge de-waxed shell were examined before and after autoclave retreatment (figure 4). The values reported in Table 8 show a slight reduction in thickness and width following autoclave treatment. However, the standard deviation is sixty times more than shrinkage difference observed and thus the result is not significant. This means that the external pressure exerted on the surface of the shell is not sufficient to show any geometry changes that could contribute to the increase of shell strength. The skeletal densities measured using Micrometrics AccuPyc ii 1340 gas pycnometer before and after autoclaving are shown in Table 8. This shows that there is no material loss from the solid phase and the mass of shell is constant.



Figure 4- The width and thickness of each sample is noted with line to assure the same location is being measured.

Table 8- The thickness and width of shell is being compared before and after the autoclaving treatment.

Shell	Before autoclaved	Standard deviation	After autoclaved	Standard deviation
Width(mm)	23.79	0.42	23.71	0.51
Thickness(mm)	6.12	0.64	6.11	0.63
Skeletal Density ($\text{g}\cdot\text{cm}^{-3}$)	2.60	0.00	2.60	0.00

Table 9- BET data of dried silica sol before and after autoclave treatment.

	BET surface area ($\text{m}^2\cdot\text{g}^{-1}$)	Langmuir surface area ($\text{m}^2\cdot\text{g}^{-1}$)	Average pore diameter by BET (nm)
Room Dried Silica Sol	179.23 \pm 0.52	247.68 \pm 8.78	5.09
Autoclaved treated Silica Sol	31.48 \pm 0.14	43.18 \pm 1.10	21.28

It is believed that the shell mechanical strength is increased by curing the silica sol in the autoclave environment rather than by structural compression. The silica sol used in slurry has a mean particle size of 14 nm and this will lead to very small inter-particulate pores if there is no coalescence. The surface area of the pores was determined using the nitrogen gas adsorption method (Micrometrics ASAP Model 2010) and the results are shown in Table 9. It can be seen that the average pore diameter of the silica sol has increased by four times after the autoclaving treatment. Autoclave heat treatment gives greater coalescence of the gel indicated by a reduction in the total surface area with larger pores forming while pore volume is retained [9]. However, autoclaving the gel network will not change the shape of gel framework, this is a common feature of gels as they become aged [9]. The resultant coalesced structures are not easily broken down when subjected to compaction. The pores in the silica gel grow larger to a point where the visible light refracts in the pores and

reduces light transmission causing the gel to appear opaque (Figure 5). The higher the degree of coalescence of the particles in the powder aggregates, the harder the aggregates will be, and the less they are deformed by mechanical stresses [10]. The X-ray diffraction (XRD) results given in Figure 6 indicate no composition change following autoclaving the silica sol. This shows that autoclaving the silica sol purely changes the pore diameter and surface area of gel. It can be concluded that it is coalescence which increases the shell strength on autoclaving. While this phenomena is most clearly seen when comparing the fridge de-waxed samples before and after autoclave treatment but the strength is also influenced by the remnant and penetrating wax.

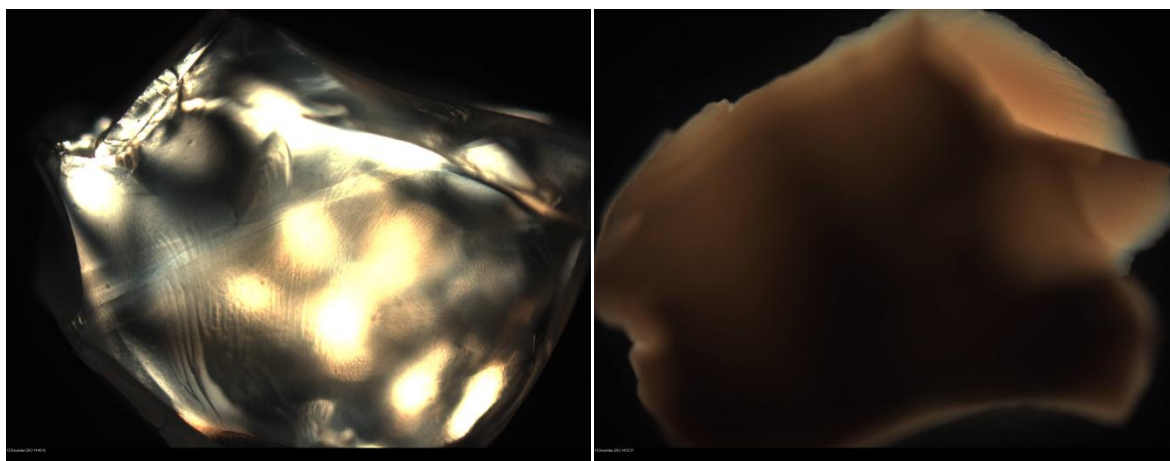


Figure 5-A transmitted light microscope was used to observe the silica sol particle dried at ambient condition (left) and silica sol cured in autoclave (right). Particle size is approximately 1mm.

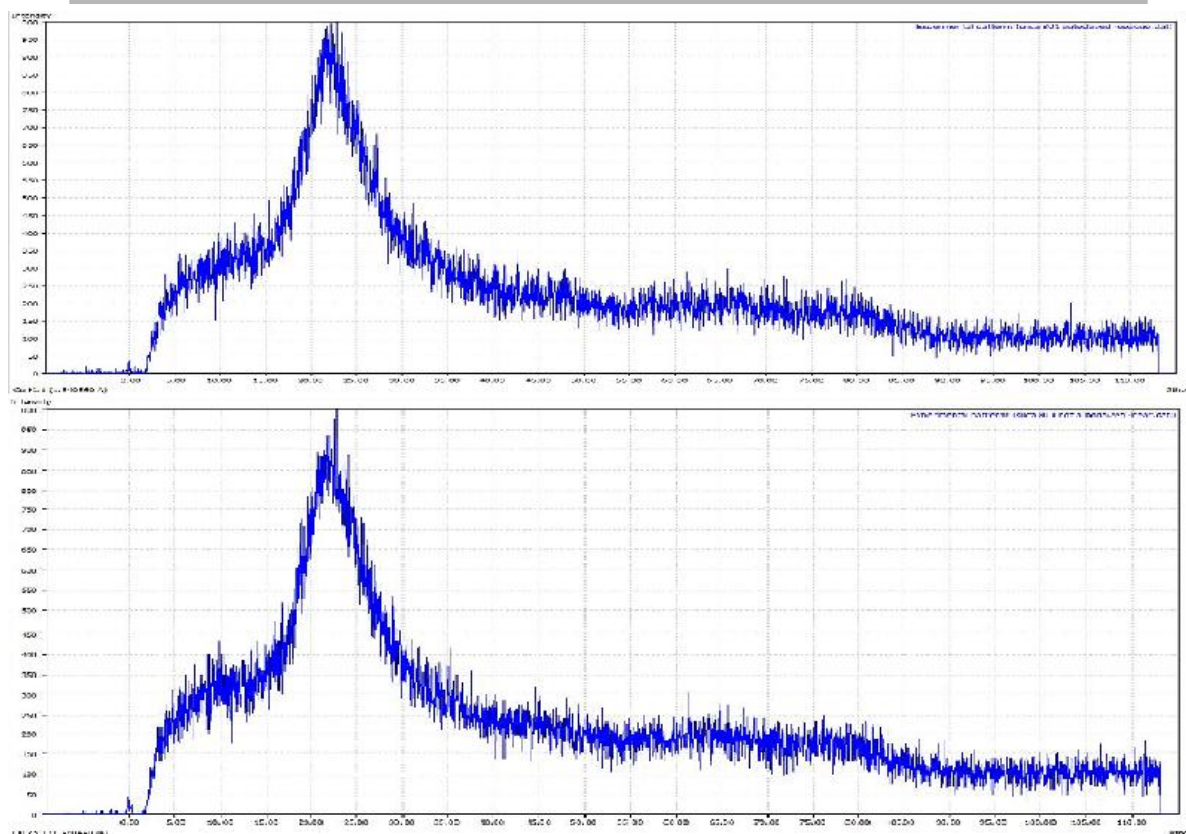


Figure 6- XRD result comparing before (top) and after (bottom) autoclaving the silica sol. It shows silica sol remain as amorphous silica.

From the results discussed in part 1 and 2, it becomes clear that it is important to determine which stage of de-waxing process is of interest in the determination of strength whether that is before or after the wax interacts with the shells. In the case of predicting de-wax shell failure, obtaining a lower limit of shell mechanical strength would perhaps be more representative of the process. The autoclaving condition is found to increase the mechanical strength of the shell as the silica sol in binder system coalesces and there is a contribution of wax permeating the shell. Tests using fridge de-waxing followed by autoclave and testing wet (method 6) produce the most useful result, as the shell is clean and less damaged from sample preparation. This more truly represents the fundamental strength of the shell system in the green state at the point of de-wax.

Conclusion

It has been shown that wax penetration into shell and the autoclave environment will affect the mechanical flexural strength of the shell. De-waxing by autoclave cures the sol by coalescence adding strength to the dried but green shell. The process of de-waxing by this method leaves some residual wax on the surface of the face coat or penetrating into the shell. De-waxing by chilling gives a clean but relatively weak shell in comparison to standard autoclave de-waxing. Treating these materials with pressurised steam increases the

strength but only to 73% of the autoclaved de-waxed which suggests that the additional strength comes from wax residues remaining in the conventional process.

For modelling autoclave dewaxing the strength of the shell should range between two values. The lower value should be that determined by dewaxing the test component by refrigeration. This represents the strength of the shell at the start of the dewax process. The upper value should be that measured on samples first fridge dewaxed and then autoclaved. The sample should then be examined while still wet. This represents the shell properties when in the autoclave at the end of the dewax process when the remnant wax would be in a molten state and contribute little to the shell strength.

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