Refractory Materials Used for the Investment Casting Process and their Influence on Ceramic Shell Properties

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Today, many different metals are being produced using the Investment Casting (IC) process. To ensure final high quality castings it is necessary to know the influencing parameters of all the different steps during the whole process including the wax pattern, the ceramic shell and the final casted part.
The research work described within this paper is about the ceramic shell, the parameters that need to be taken into account during the whole processing and the possibilities to evaluate and influence them in a proper way. The main parameters discussed are the casting dimensional accuracy, the hot properties of the shell and the shell building performance itself.

1 Introduction

The investment casting process is used for high precision castings. A wax model of the final casted part is prepared and afterwards dipped into a slurry based on binder, refractory filler and additives which are influencing the wetting and foaming behaviour of the slurry.
Afterwards, so called stucco material is applied on the wax coated with the slurry and the combination of both, slurry and stucco, build up the first layer of the shell. When the layer is completely dried, this process is repeated until the desired thickness of the shell is reached, normally around 6–12 layers depending on the size of the casted part and the casted metal/alloy. However, the slurry as well as the stucco is different for different layers. The first layer is in direct contact with the metal and therefore mainly responsible for the quality of the surface of the casted part. This layer needs to be fine in particle size distribution (~100–200 µm) and high in refractoriness. The next layer is called intermediate layer and is responsible for connecting the first layer with the back-up layers. Also this layer sees a relatively high temperature, although it is not in direct contact with the casting.
Therefore, the particle size distribution can be slightly coarser (~200–500 µm) due to the fact that it is not anymore directly responsible for the surface smoothness. The stucco for the backup layers are relatively coarse in particle size distribution (~0,5–1 mm) and often a less refractory material can be used (e.g. chamotte), depending on the casting process. These layers build up the final thickness of the shell and are mainly responsible for the mechanical strength.
After the shell has been build up the wax needs to be removed, which is normally done in an autoclave. Prior to the casting, the shell is heated up to remove the organics and to minimise the thermal shock during the casting process. In the next step the metal is poured inside the shell and the cooling process starts. In the final step the ceramic shell is removed from the casting (knock out) and the part is further processed (e.g. cutting, sand blasting, grinding, polishing).

2 Raw materials for the investment casting process

Generally several different raw materials are used, depending on the alloy, the casting temperature and the layer where the material is applied. All of the refractory raw materials have a specific mineralogical composition comprising one or more phases, depending on its paragenesis and its manufacturing process. During the process the phase composition of the material may change due to the temperature and time cycle according to occurring phase transformations.
The main groups of refractory raw materials are either natural or synthetic and the latter includes calcined, sintered or fused products. Sintered/calcined materials are e.g. mullite, kaolin, chamotte, tabular alumina, zircon. Fused raw materials are e.g. alumina, mullite, silica, yttria and stabilised zirconia (Fig. 1).

2.1 Basics on particle dispersion and particle size distribution

Investment casting slurry comprises a large amount of fine particles which tend to agglomerate but also coarser particles need to be prevented of sedimentation in the slurry. This means appropriate dispersion in the investment casting slurry tank has to be guaranteed with good tank size, special designed blade and correct rotation speed. Generally speaking, rheological behaviour of investment casting slurry is controlled by particle size distribution, shape of particles, solid loading and liquid component.

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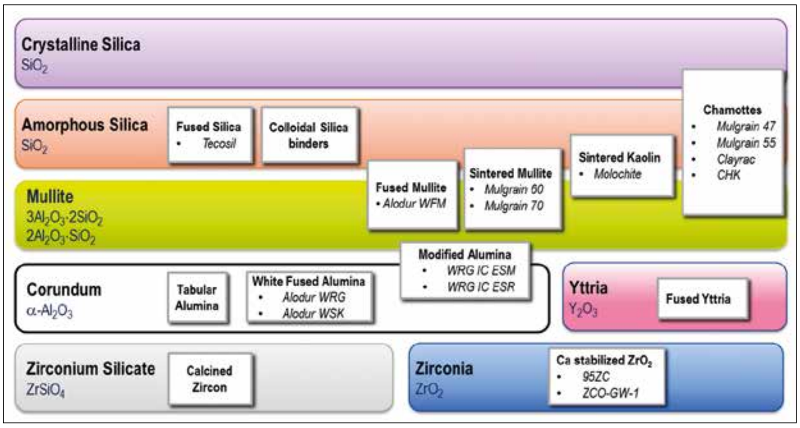

Fig. 1 **Overview of different phase compositions used for investment casting and the chemistry thereof**

All these parameters have to be adjusted to finally obtain a casting shell structure with enough strength to withstand casting forces but also it has to be permeable enough to minimise casting defects.

There is not one optimal solution suitable for all casting process. It is more about finding the perfect compromise for each individual process [1].

2.2 The use of alumosilicates, fused silica and blends thereof

Fused silica is used in IC-Shells because of its very low thermal expansion and of its great shell removal properties. Especially, the de-vitrification of fused silica into cristobalite and the phase transformation from β- into α-cristobalite at 200–300 °C when the shell cools again have powerful influence on the shell properties [2].

Alumosilicates in investment casting application vary in a wide range. On one side of the scale artificially fused ones with 100 % mullite and nearly no impurities to the other side more impure materials rich in alkalis and glass phase. However, all these numerous different materials have its advantages and disadvantages looking at chemical stability, price, thermal stability or strength giving to the shell.

Recently developed blends of different alumosilicates with fused silica are able to give specially designed properties for specific applications as alumosilicates and fused silica show a strong influence on each other. Physical properties as well as mineralogical composition change significantly during the investment casting cycle.

Especially the influence of alumosilicates on time and amount of cristobalite formation during the thermal cycle is very strong (Fig. 2). In this respect Imerys has developed new investment casting flour called HYBRID+.

Summarised, most alumosilicates in connection with fused silica lower the cristobalite formation significantly (up to –80 %). Nevertheless this does not influence the shell removal properties too much what can be clearly seen in the Cold Modulus of Rupture (CMOR) trend of different blends (Fig. 3). High CMOR values after firing, which are typical for pure alumosilicates, indicate hard shell removal.

Whereas quite low CMOR values after firing which are typically for mentioned blends can be correlated with easy shell removal properties. The impact on strength of fused silica on easy shell removal varies in between different alumosilicates. However, the trend to an easier shell removal is always the same.

Blending fused silica to different alumosilicates has quite diversified impacts on hot deformation stability. On the one hand, hot deformation can be extremely improved and on the other hand blending can weaken these properties (Fig. 4). The secret behind all these very different behaviours is the supporting or suppressing effect of cristobalite, mullite and glassy phase formation which is unified in the new flour HYBRID+.

2.3 Chemical stability of key raw materials

As contact layer material, zircon is historically widely used in investment casting application. It is known to give very good performance looking at natural occurring raw materials. Nevertheless, natural zircon meets its limits when customer ask for castings in very reactive alloys, increased part sizes or more and more reduced defect rates. In this connection most common reasons for increased scrap rates are zircon

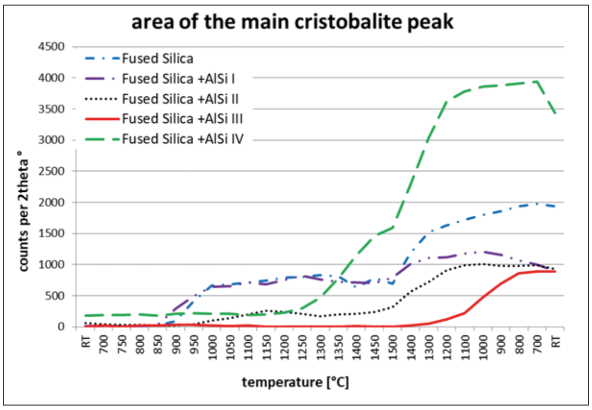

Fig. 2 **Cristobalite formation during a HT-XRD measurement what simulates a casting cycle – beside the pure fused silica sample, all blends contain at the beginning 75 % fused silica, 25 % alumosilicate and a Na-stabilised Si-binder**

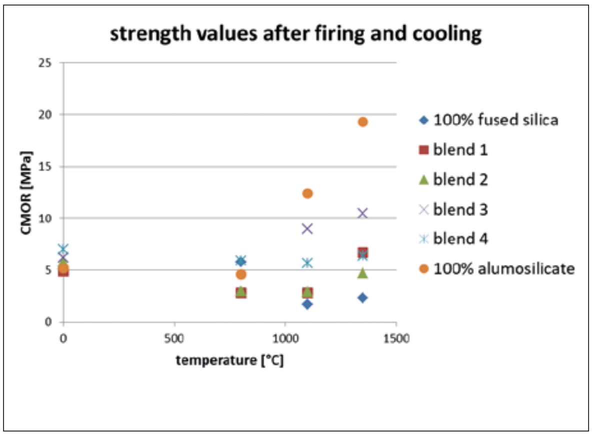

Fig. 3 **Cold modulus of rupture values of tested blends, showing the benefit of fused silica-alumosilicate blends concerning easy shell removal**

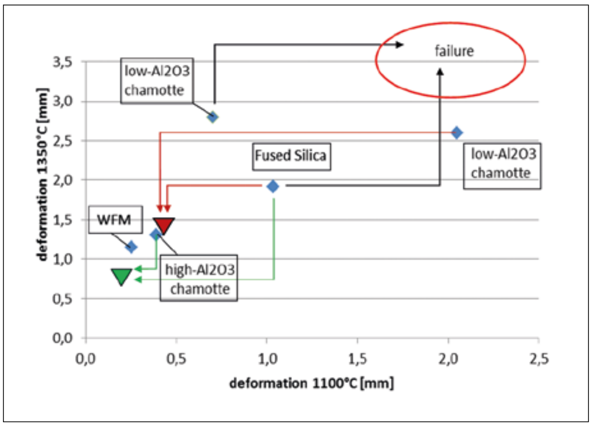

Fig. 4 Hot deformation properties of pure materials and blends thereof with fused silica

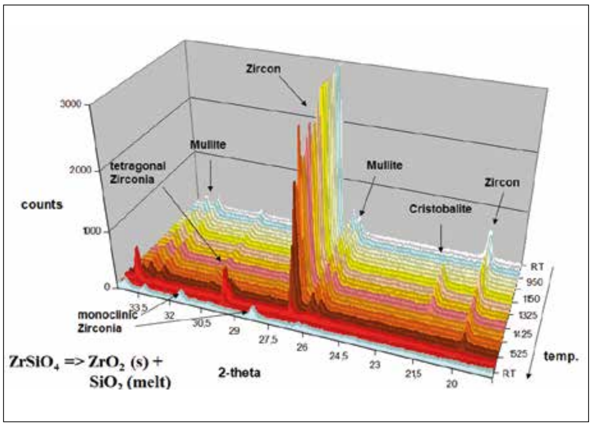

Fig. 5 High temperature XRD showing phase development of the initial composition: zircon flour, corundum flour and silica glass from the binder (no peaks); also zircon decomposition into ZrO_2 and SiO_2 (melt – no peaks) is visible

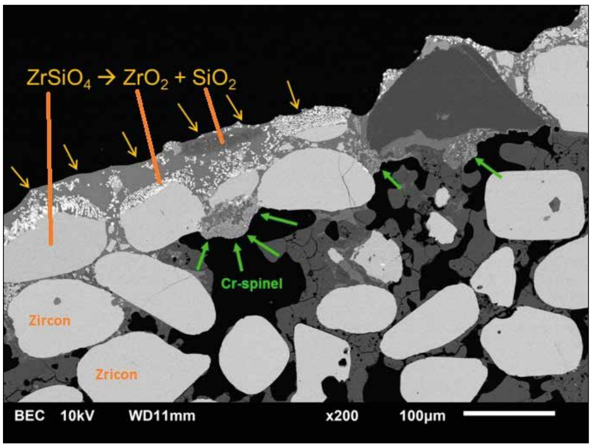

Fig. 6 Zircon ($ZrSiO_4$) decomposition to ZrO_2 and liquid SiO_2 during casting in comparison to a casting with modified alumina in contact to metal without reactions in contact area (Fig. 7)

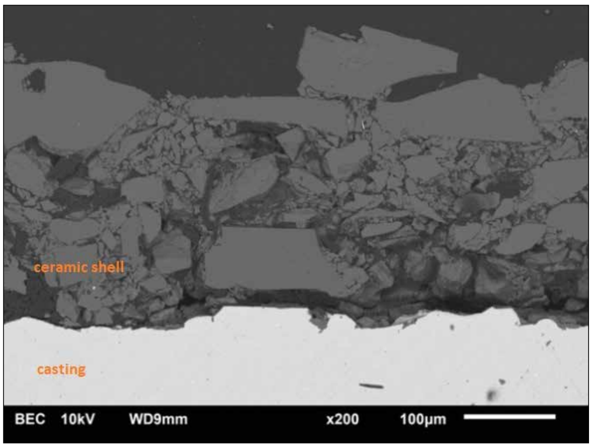

Fig. 7 In comparison to Fig. 6 (chemical reaction between ceramic shell and metal): ceramic shell with modified alumina in contact to metal after casting without reactions in contact area

decomposition and reactions with impurities in zircon raw material. (Fig. 5–6). Of course, process with adjustment of slurry rheology, dipping procedure, drying, applying stucco or firing are critical as well but should not be focus of this paper. Summarized, synthetic raw material as basis for whole process can bring defect rates on a new level (Fig. 7).

For backup layers usually different qualities of alumosilicates are used as flour in the slurry as well as stucco due to perfect price-performance ratio. Development of mineralogical phases during the firing and casting process are key for sufficient strength, good permeability and hot deformation resistance. In this connection the formation of mullite, glassy phase and creation of right sized porosity are critical. Beside diverse alumosilicates also fused silica and for special application like single crystal also special modified fused alumina gets used.

2.4 Synthetic raw materials for highly reactive alloys

The use of yttrium-oxide or zirconium-oxide as a refractory flour in the face-coat of the investment casting mould is the best way to protect highly reactive and expensive metals from chemical reactions with the ceramic shell.

The benefits of fused yttrium-oxide or fused zirconium-oxide are mainly related to the extremely high refractoriness, very high affinity to oxygen, coupled with high purity and low porosity, which results in mould face-coat particularly stable whatever is the casting temperature and alloy.

Titanium and titanium aluminide are the most common metals used together with yttrium-oxide or zirconium-oxide as this combination offers significant advantages for production. These advantages are the possibility to produce near net shape castings with good surface quality and therefore – although such materials are quite expensive – still it offers best price-performance ratio.

Special shell moulding system designed for the use as face-coat for high temperature alloys such as titanium or titanium aluminides are available on the market. The primary layer(s) of these shells are composed of fused yttrium-oxide filler and fused yttrium-oxide or fused alumina stucco. The fused yttrium-oxide is used together with a special alumina binder. This binder finalizes the great properties of fused yttrium flour in investment casting application.

Tab. 1 Example for shell composition

Coat	Filler	Binder	Stucco
Primary coat	Fused alumina Treibacher Alodur® WRG IC ESM	Compatible with a wide range of binders e.g. different Na-stabilised binders	Fused alumina Alodur® ZWSK
Secondary coat (necessity depends on casting geometry, slurry rheology and casting process)	Fused alumina Treibacher Alodur® WRG IC ESM	Compatible with a wide range of binders e.g. different Na-stabilised binders	Fused alumina Alodur® ZWSK
Back-up coats	Wide range of materials possible including chamottes, sintered or fused mullite and HYBRID+; all should be adjusted with primary coat		

Tab. 2 Each step during the shelling process is responsible for the final casting quality, the raw materials used, the slurry prepared thereof, and the final ceramic shell made from the slurry and the stucco used

Chemical Properties		
Chemical composition	→	X-Ray Fluorescence spectroscopy (XRF)
Mineralogical composition	→	X-Ray Diffraction (XRD; Rietveld refinement)
Slurry pH	→	pH-meter
Slurry stability	→	Zeta potential
Physical Characteristics		
Shell strength and delamination	→	Bending strength measurement
Gas permeability, porosity	→	Airflowmeter, Hg-porosimeter
Dimensional changes	→	Dilatometry
Slurry viscosity, plate weight	→	Viscometer, flow cups, balance
High Temperature Performance		
Refractoriness	→	Refractoriness under Load (RuL)
Creep	→	Creep in Compression (CiC)
Thermal conductivity	→	Laserflash (LFA)
Mechanical strength	→	Hot-MOR
Shell Structure		
Shell structure	→	Optical microscope
Explaining of structure-related casting defects	→	Scanning Electronic Microscope (SEM, EDX)

Fig. 8 Shelled ping pong balls for determining the permeability of the shell

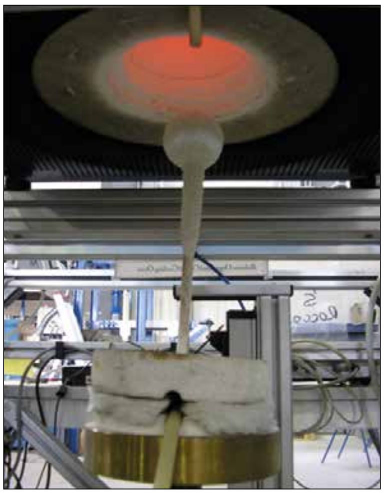

Fig. 9 Furnace for measuring the hot permeability

2.5 Synthetic raw materials for enhanced performance

The use of fused alumina as a refractory flour in the face-coat of the IC mould is more and more frequent, particularly as an alternative to zircon for steel industry or producers of super alloy castings. The benefits of fused alumina are mainly related to the high refractoriness, coupled with high purity and low porosity, which results in mould face-coat particularly stable whatever is the casting temperature. One key in this connection is also the reaction with SiO_2 in the binder to a more stable phase during firing.
As a consequence, the introduction of fused alumina significantly reduces the metal-mould reaction and eliminates surface defects which result from the impurities that are always associated with zircon (calcined or not). Through a modification of the original fused alumina, its properties could be enhanced further particularly regarding shell removal and part cleaning.
Tab. 1 shows a general example of such a shell system based on fused alumina front layer. Also this shell system may need to be adapted slightly by individual foundries to suit their product range and casting methods.

3 Controlling of shell parameters

Good process control is achieved through close inspection of all stages during the shell building process. Tab. 2 shows which parameters at each step need to be contemplated.
Parameters which need to be taken into account and the method of choice for determining them are shown in the following paragraph and some of the more critical parameters will be further discussed.

3.1 Permeability of the ceramic shell

The gas permeability has a significant influence on the final casting quality. To determine the shell permeability a ping pong ball is attached to a ceramic tube and shelled (Fig. 8). Afterwards, the ball is burned out. This process needs to be controlled carefully to avoid a cracking of the shell due to gas pressure coming from the plastic ball during the combustion process. Afterwards, the ceramic shell can be sintered and an air flow meter is attached to the ceramic tube. The pressure is kept constant and the airflow is measured in [l/min]. The authors

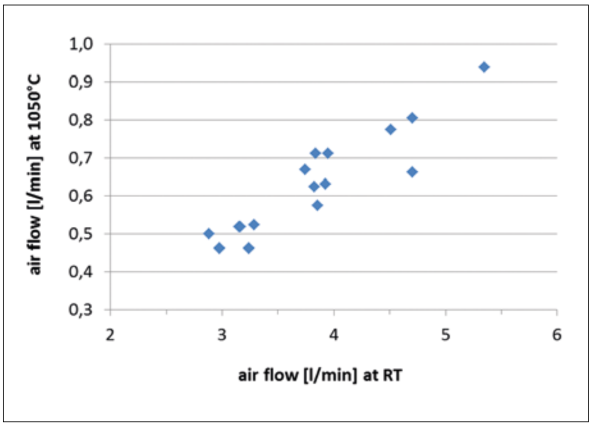

Fig. 10 Permeability measured at room temperature compared to the permeability at 1050 °C

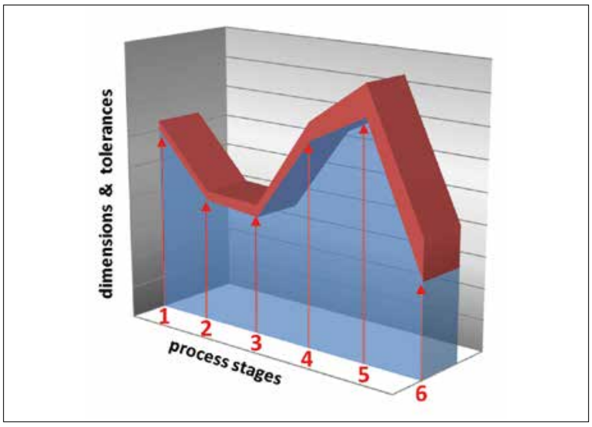

Fig. 11 Dimensional changes throughout the IC process [3]

designed a special furnace up to 1600 °C which has a hole in the bottom, as shown in Fig. 9. Due to that, the ceramic ball can be placed inside the furnace and the airflow can still be measured through the ceramic tube. This allows measuring the hot state permeability, which is a good simulation of the permeability during the casting process. Due to sintering and expansion effects the pores are closing and thus, the permeability is lower at higher temperature compared to room temperature. Nevertheless, in several cases the permeability at room temperature can be correlated with the permeability at higher temperatures, as seen in Fig. 10. These shells were based on molochite with different particle size distributions and tested at room temperature and 1050 °C respectively. One can clearly see a linear trend between those two measurements.

3.2 Dimensional accuracy

The dimensional accuracy for the final casting plays an important role and therefore it is important to consider all the dimensional changes occurring during the different steps. As shown in Fig. 11, the first shrinkage occurs from the hot wax injection in the tool cavity (1) to the cooled wax pattern (2). A study on the dimensional changes of the wax during the processing was done by Bond et al. in 2012 [3].

During the shelling itself (3), the size stays constant, but increases during the preheating (4) due to thermal expansions of the used raw materials. A second expansion of the shell happens during the pouring of the liquid metal caused by the temperature increase of the material (5).

Finally, the cooling of the metal is resulting in a dimensional shrinkage (6) till the dimensions of the final casting is achieved. Sabau showed in his study from 2006 how to predict the contribution of the alloys to the final part dimension [4].

The thermal expansion of the raw materials needs to be taken into account for the calculation of the pattern size to achieve the right dimensions for the final castings. This is mainly a sum of the expansion coming from the flour and stucco used during the shell building process. The phase composition and its modification due to the heating of the shell are influencing this parameter. Fig. 12 shows the thermal behaviour of several typically used stucco materials.

As one can see, the fused silica obviously shows nearly no expansion at all due to its nature of being amorphous, and therefore this material exhibits a good dimensional accuracy. Due to the presence of cristobalite, the chamotte has a strong expansion around 200 °C.

This is caused by the transformation of the α-phase (true density: 2,32 g/cm^3) into the β-phase (true density: 2,20 g/cm^3) and the related volume increase of approximately 5 % [2, 5]. The thermal expansion curves also indicate a softening of a sample and a re-stabilisation due to the transformation of aluminiumoxide and silicium oxide into mullite.

3.3 High temperature performance

The measurement of the creep in compression gives an idea of the shell performance under the pressure of the molten metal casted into it. For this test, a shell with 5 cm diameter and the same height is build up around a cylindrical wax part. Afterwards the

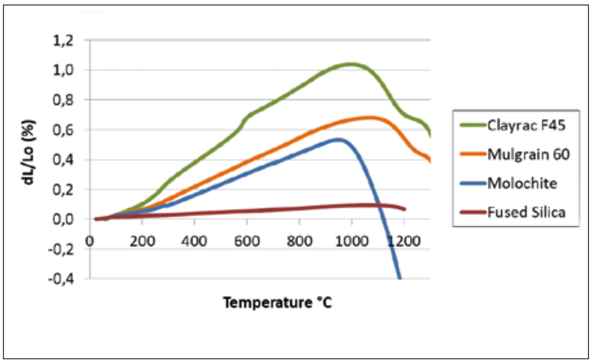

Fig. 12 Thermal expansion of different IC raw materials

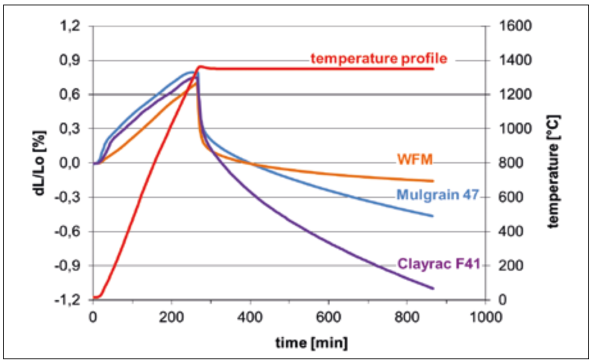

Fig. 13 High temperature performance of different shells under load

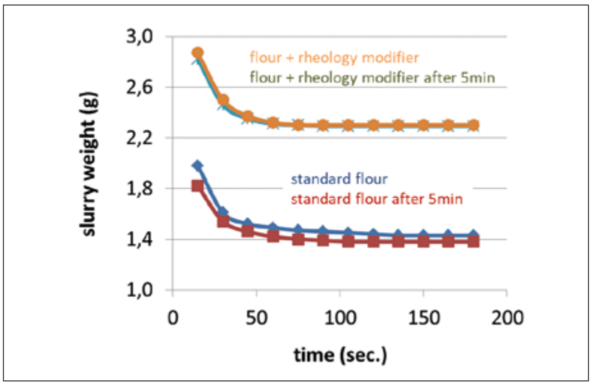

Fig. 14 Plate weight of an IC slurry based on a standard flour and a modified one

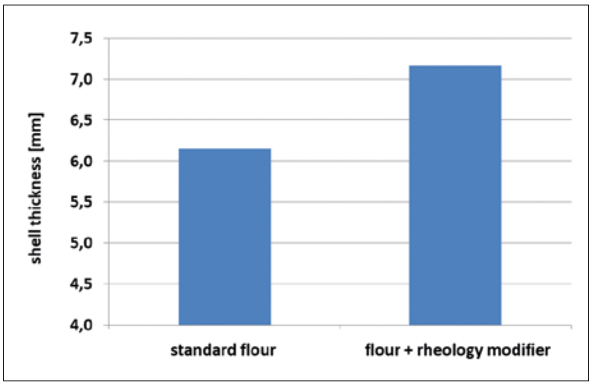

Fig. 15 Shell thickness after applying 5 layers

wax is burnt out to have a shell in the typical cylindrical shape for measuring the creep in compression. Due to the lower green strength of the shell, the weight is applied on the sample after the targeted temperature (in this case 1350 °C) inside the furnace is reached.

Therefore a first significant shrinkage can clearly be seen, which is caused by the weight addition. After this initial creep, the continuous deformation of the shell under load is recorded over the time. In Fig. 13, the results of the investigations of three different shell systems are shown. One shell was based on white fused mullite, another one was based on Mulgrain 47, which is a sintered mullite with an alumina content of around 47 %, and the last sample is a chamotte with an alumina content of 41 %, called Clayrac F41. One can clearly see the differences in the stability of the three specimen. Due to a certain amount of cristobalite inside the Mulgrain 47 and the Clayrac 41 sample, a thermal expansion of the shell around 200 °C during heating up can be observed. The shell based on white fused mullite shows the highest creep resistance at 1350 °C. The creep rate between 2–10 h for WFM was –0,02 %/h.

The Mulgrain sample showed a creep of –0,06 %/h, and the Clayrac F41 –0,11 %/h. This can be used as an indicator for the resistance of the shell during the casting process and gives a clear picture that it is from great importance to choose the right shell materials for the targeted casting process.

3.4 Shell building performance

For characterising the shell building performance, the so called plate weight test is used. For this, a thin metal plate with a well-defined length and broadness is dipped inside the slurry for 30 s. The plate is than hung onto a balance where the dripping slurry is caught on a separated petri dish, so that it is not directly influencing the weight on the balance itself. With this procedure, it is possible to determine the weight of the slurry that is wetting the surface of the plate and its draining behaviour in dependence on time. Fig. 14 shows the plate weight of a standard aluminiumsilicate flour in blue.

The slurry is rested for 5 min, and then a second measurement is done (red curve). This gives an idea of the sedimentation behaviour of the material. If both curves are close together, the sedimentation of the slurry is low. A high sedimentation rate would give a higher difference between the plate weight of the two cycles. Additionally, it can be seen from Fig. 14 that the filler load can be increased by adding rheology modifiers into the slurry, which has a direct influence on the plate weight.

Fig. 15 shows the shell thickness of a standard flour compared to a modified one. Both shells are based on 5 layers. One can see that the modified shell was more than 7 mm thick whereas the standard shell was around 6 mm. Due to the higher filler load, the plate weight is higher and therefore the shell thickness is increased. This means that fewer layers need to be applied to reach a certain thickness of the final shell.

4 Conclusions

The investment casting process is quiet complex, and therefore a lot of different parameters can influence the final casting quality. One important step is the shell building and the material used for it, which has to be decided due to the requirements of the metal or alloy that will be casted, as well as the price of the materials used. Many different factors of the raw materials have to be taken into account e.g. chemical composition, phase composition or thermal expansion.

These properties define the performance during the casting, the influence on the dimensional accuracy, the permeability of the shell and the mechanical stability to withstand the casting. Also an easy shell removal after the casting, which can be achieved due to volume increasing phase transformations and therefore soften the shell, are from importance. Understanding, determining and controlling all of these steps are one main part in order to successfully perform high demanding castings.

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