www.czasopisma.pan.pl PAN www.journals.pan.pl

ARCHIVES
of
FOUNDRY ENGINEERING

DOI: 10.1515/afe-2017-0136

Published quarterly as the organ of the Foundry Commission of the Polish Academy of Sciences

ISSN (2299-2944)
Volume 17
Issue 4/2017

91 – 96

Influence of the Al_2O_3 Solid Phase on the Kinetics of Binding Ceramic Moulds

J. Kolczyk *, J. Zych, Ł. Jamrozowicz
AGH University of Science and Technology, Faculty of Foundry Engineering,
Department of Moulding Materials, Mould Technology and Cast Non-Ferrous Metals,
Al. Mickiewicza 30, 30-059 Kraków, Poland
* Corresponding author. E-mail address: kolczyk@agh.edu.pl

Received 22.06.2017; accepted in revised form 15.09.2017

Abstract

The investigation results of the kinetics of binding ceramic moulds, in dependence on the solid phase content in the liquid ceramic slurries being 67, 68 and 69% - respectively, made on the basis of the aqueous binding agents Ludox AM and SK. The ultrasonic method was used for assessing the kinetics of strengthening of the multilayer ceramic mould. Due to this method, it is possible to determine the ceramic mould strength at individual stages of its production. Currently self-supporting moulds, which must have the relevant strength during pouring with liquid metal, are mainly produced. A few various factors influence this mould strength. One of them is the ceramic slurry viscosity, which influences a thickness of individual layers deposited on the wax model in the investment casting technology. Depositing of layers causes increasing the total mould thickness. Therefore, it is important to determine the drying time of each deposited layer in order to prevent the mould cracking due to insufficient drying of layers and thus the weakening of the multilayer mould structure.

Keywords: Innovative foundry technologies and materials, Investment casting technology, Ultrasonic tests, Ceramic moulds, Aqueous binders

1. Introduction

A ceramic mould is formed as a result of depositing a few layers of the liquid ceramic slurries on the wax model. Each layer is sprinkled by a high-refractory material used as a powder topping [1, 2]. Ceramic moulds are not homogeneous materials. Properties of multilayer ceramic moulds depend - to a high degree - on initial materials used for their production. A binder together with a high-refractory matrix constitutes the liquid ceramic slurries. In dependence of the kind (mullit, Al_2O_3 – pure technical aluminium oxide, silimaniat, etc.), amount of matrix, grain size of the applied material, various dynamic viscosity values of ceramic slurries are obtained [3, 4]. Deposition of individual layers is a multistage process (from 4 to 8 layers). When the successive layer is deposited the previous one should be properly dried [5, 6] since otherwise moisture from the mould depth will not be removed and the mould strength will be lowered. Therefore, the drying time determination of individual layers of the ceramic mould, by means of non-destructive tests, is the important element of the technological process.

There is a clear tendency in the foundry industry to introduce investigation systems, in which foundry materials as well as castings are controlled by various non-destructive methods. A non-destructive quality control using ultrasound, radiographic, magnetic, centrifugal or penetrating method is applied at various production stages. A progress in constructing scientific research equipment allows obtaining more information and a higher precision of measurements. This widens research possibilities by applying individual non-destructive tests. By ultrasonic tests it is possible:

- to determine the metal quality thus before its pouring into the mould,
- to assess mechanical properties,
- to assess the structure in the selected place of the casting,
- to perform a segregation of castings due to their structure,
- to detect and assess internal defects of castings (quality control) [7].

Propositions of using ultrasonic waves for investigations slurries appeared in the foundry engineering some years ago [8, 9]. In 2006 Zych [10] patented the new method of investigating the hardening process of slurries with binders by applying the ultrasonic technique. This created new possibilities for monitoring the binding processes of slurries (Patent PL 192202 B1). He indicated in his research that - by means of ultrasounds - it is possible to determine the apparent density of humid moulding sands and their compaction degree in the mould, to investigate the condensation zone and kinetics of its formation as well as to investigate effects occurring in surface layers of sand moulds. Zych performed also ultrasonic investigations aimed at monitoring the hardening process of moulding sands with chemical binders as well as the binding and hardening processes of materials with cement binders [11]. The author described in detail the methodology of investigating ceramic materials [12], using the phenomenon of waves propagations in multilayer zones built of layers being in parallel to the wave propagation direction.

2. Ultrasonic investigations – measurement methodology

The device being in the Laboratory of Foundry Moulds Technology of the Faculty of Foundry Engineering AGH was used for assessing the kinetics of drying liquid ceramic slurries by means of the ultrasonic technique.

The probe shown in Figure 1 was used for investigations the hardening process of the multilayer ceramic mould. After depositing the ceramic layer, during its drying period the probe was placed in the measuring chamber (Fig. 1a) of the stabilised temperature and air humidity.

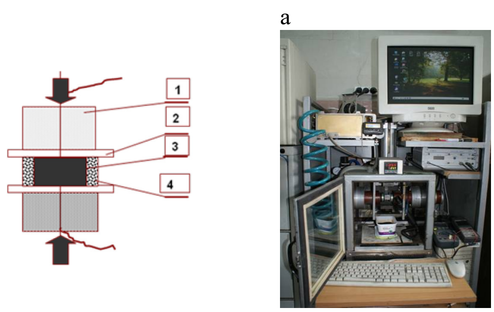

Fig. 1. Idea of ultrasound investigations of thin-layers hardening (a) and the measuring stand for ultrasonic investigations in the air conditioned chamber [10]

Successive layers of the ceramic slurries (4) were deposited on the sample core (3) and sprinkled additionally with Mullit II. After depositing of each layer the probe was placed in the measuring chamber between heads (1). Tests were performed at a temperature of 25°C. During the whole testing time the sample was in the probe, which was placed between two ultrasonic heads cooperating with the materials tester. In order to force the ultrasonic waves propagation and to create conditions for measuring wave velocity in the ceramic layer, the sample core (3) was made of the material strongly damping the ultrasonic wave. The wave is transferred via two zones, but the receiving head records arriving of the first signal, it means the one which was reaching the head by transferring through the mould layer. The measurement was based on transmitting ultrasonic waves and recording signals every 60 seconds. The investigations were performed in order to determine the drying velocity (pathway) of successively deposited mould layers.

3. Kinetics of drying (hardening) of individual layers of the ceramic mould

The velocity of the ultrasonic wave c_L transmitted by the ceramic mould layer was determined according to the simple dependence (1).

$$c_L = \frac{d}{t} \tag{1}$$

where:
c_L – velocity of a longitudinal wave, m/s,
d – distance of the measuring zone from the head, m,
t – time of the ultrasonic signal transmission through the sample, s.

From the very beginning of applying new aqueous binders the main technological problem constitutes „forcing" of relatively fast drying of individual layers. Due to a possibility of deforming wax models at increased temperatures, drying must be carried out at relatively low temperatures (24÷25°C). It is important to recognise the influence of some factors which can change drying pathways of successive layers. To such factors belongs: layer thickness, grain composition of a matrix for sprinkling successive layers, air movement above a drying layer and others.

Investigations of drying individual layers were performed for some ceramic slurries based on the Ludox AM and SK binders. These slurries differed in the solid phase content, which was equal: 67, 68, 69%, respectively. Investigations concerned monitoring of the drying process of successive three layers of ceramic slurries with Mullit II matrix. A temperature inside the chamber was 25°C. Examples of drying pathways of the individual ceramic mould layers made of slurries containing 67, 68 and 69% of the solid phase (Ludox AM and SK) are presented in Figures: 2 to 7.

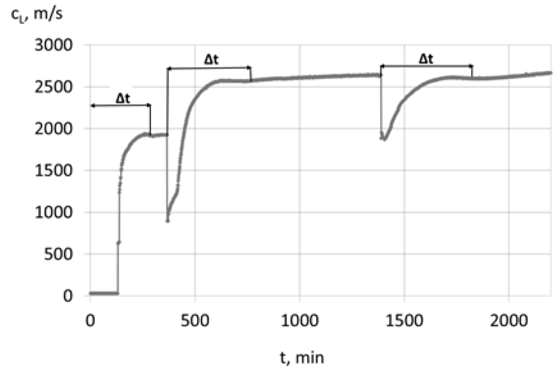

Fig. 2. Hardening pathways of three successively deposited layers of the ceramic mould (with Mullit II matrix), Ludox AM binder (solid phase content –67%)

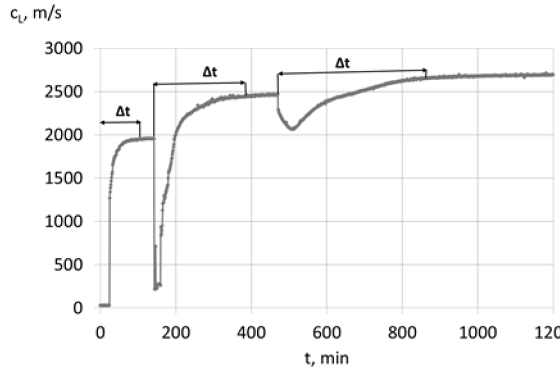

Fig. 5. Hardening pathways of three successively deposited layers of the ceramic mould (with Mullit II matrix), Ludox SK binder (solid phase content –67%)

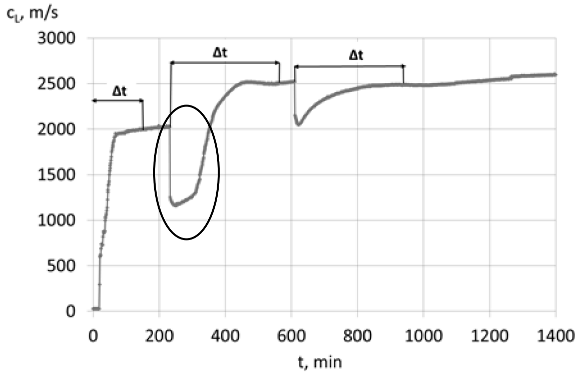

Fig. 3. Hardening pathways of three successively deposited layers of the ceramic mould (with Mullit II matrix), Ludox AM binder (solid phase content– 68%)

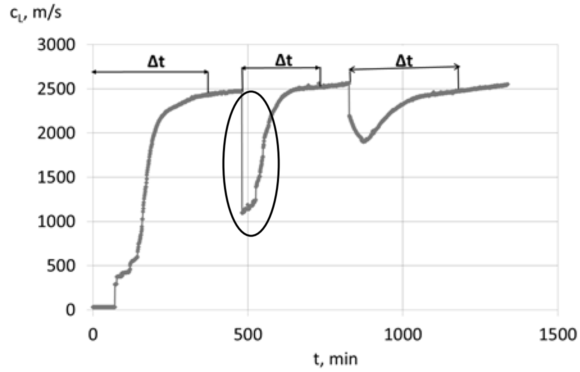

Fig. 6. Hardening pathways of three successively deposited layers of the ceramic mould (with Mullit II matrix), Ludox SK binder (solid phase content– 68%)

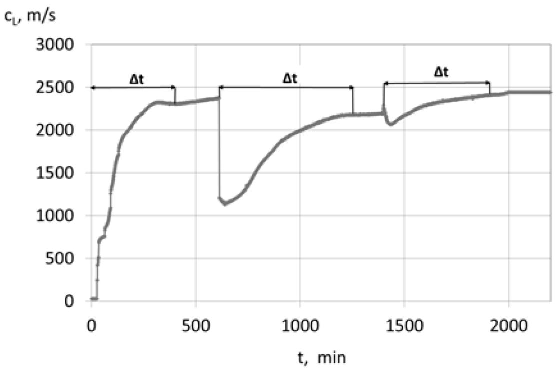

Fig. 4. Hardening pathways of three successively deposited layers of the ceramic mould (with Mullit II matrix), Ludox AM binder (solid phase content – 69%)

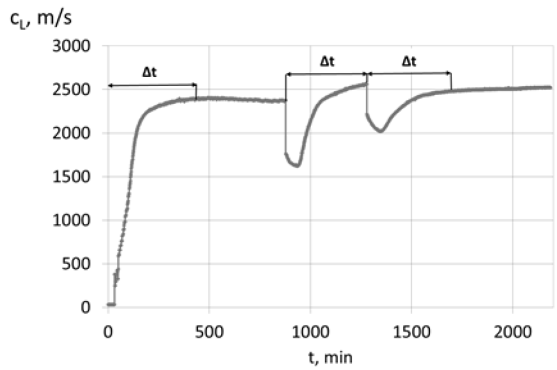

Fig. 7. Hardening pathways of three successively deposited layers of the ceramic mould (with Mullit II matrix), Ludox SK binder (solid phase content– 69%)

As far as the ceramic layer dries its ability to wave propagation increases, which leads to the observable velocity increasing. The segments presenting the drying times (Δt) of the coating are marked in Figure 3 and 6. Their determinant constituted the wave velocity, which was not changing its value at the final stage of the process.

Along with increasing the solid phase content within limits: 67÷69% the layer drying time prolongs. The longest time was - in each case - noticed for the second layer (Table 1 and 2). This is caused by the total hydration of the previously put base, which was presented in Figure 3 and 6. Soaking the previously made layer with a binder, is the reason of the increased drying time. In a similar fashion, as in case of depositing three layers of the slurries made on the basis of the Ludox AM and SK containing 68% of a solid phase, behave layers of the ceramic mould made with additions of 67 and 69 % of a matrix. In Table 1 and 2 the drying time changes of individual mould layers are presented (1, 2 and 3 layer) in dependence of the aluminium oxide percentage content and the binder kind.

Table 1.
Drying time changes of layers in dependence on the solid phase content in the liquid ceramic slurries made on the basis of the Ludox AM binder

Content of solid phase [%]	Number of deposited layers	Change of the drying time of layers Δt [h]	Entire drying time of ceramic mould [h]
67	1	4,5	**18**
	2	6,5	
	3	7	
68	1	2,5	**13,5**
	2	5,5	
	3	5,5	
69	1	6,5	**25**
	2	10,5	
	3	8	

Table 2.
Drying time changes of layers in dependence on the solid phase content in the liquid ceramic slurries made on the basis of the Ludox SK binder

Content of solid phase [%]	Number of deposited layers	Change of the drying time of layers Δt [h]	Entire drying time of ceramic mould [h]
67	1	1,5	**12**
	2	4	
	3	6,5	
68	1	6	**16**
	2	4,25	
	3	5,8	
69	1	7	**21,5**
	2	6,8	
	3	7,7	

The observed, prolonged drying time - at the increasing solid phase content - occurs due to the fact that the thickness of deposited layers increases at the increasing solid phase fraction (%), which is related to the viscosity increase of the liquid ceramic slurries.

Thus, summarising, it can be stated that the solid phase content increase in the liquid ceramic slurries causes prolongation of the layer drying time. Using the ultrasonic method it is possible to determine the ceramic multilayer mould strength at individual stages of its production. The second layer deposition causes a temporary loss of elasticity and strength of the ceramic mould, while the successively deposited layers cause longer drying time of the mould.

4. Influence of the layer thickness on the drying time of ceramic moulds

The ceramic layer thickness depends, among others, on the dynamic viscosity of the liquid ceramic mixture. Introduction of a larger amount of solid phase into the slurries increases its viscosity, which - in turn - causes the longer drying time of the ceramic mould. Therefore, an amount of the introduced solid phase into the liquid ceramic slurries are important for the mould production technology.

The plate made of the model mixture (wax) (Fig. 8) was used for assessing the influence of the layer thickness on the mould drying time. Investigations were performed for the ceramic slurries made on the aqueous binder base of the commercial name: Ludox AM and SK, of the solid phase content being 67, 68 and 69%, respectively.

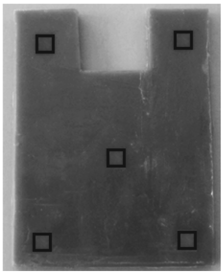

Fig. 8. Wax model formed after the matrix was poured with wax

Three layers of the ceramic slurries were deposited on the cleaned and degreased wax model. After each deposition of the ceramic slurries and drying of the successive layer the coating thickness was measured in five various points (marked as squares in the photo). Then the average thickness value was calculated.

The measurements were performed by means of the micrometer screw, of the measuring range from 0 to 25 mm and the accuracy of 0.01 mm. The investigations purpose was the determination of the influence of the Al_2O_3 phase content on the thickness of deposited layers.

Figures 9 and 10 present wax models of plates with deposited three layers of the liquid ceramic slurries on the base of Ludox AM and SK binders, with various fractions of the solid phase in the liquid ceramic slurries.

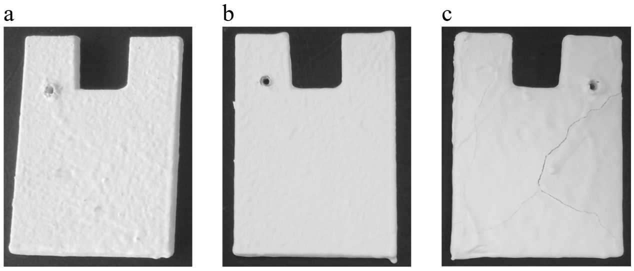

Fig. 9. Wax models after the deposition of three layers of the ceramic slurries (Ludox AM) of a various content of solid phase: a – 67% , b – 68% , c – 69%

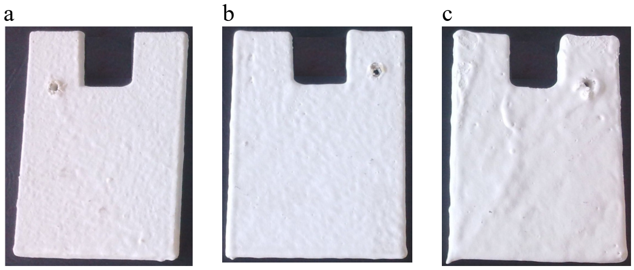

Fig. 10. Wax models after the deposition of three layers of the ceramic slurries (Ludox SK) of a various content of solid phase: a – 67% , b – 68% , c – 69%

The introduction of a small amount of aluminium oxide into the mixture (67% of solid phase) causes uneven covering of the wax model surface. The ceramic slurries layer is very thin (Fig. 9a and 10a). Whereas the introduction of too large solid phase fraction (69%) causes the dynamic viscosity increase, which leads to uneven covering of wax models by ceramic slurry. In addition, it causes formations of lumps, various irregularities of surfaces and cracks (seen in Fig. 9c and 10c). The deposition of the successive layer of the liquid ceramic mixture on the wax model causes that coatings are becoming thicker and thicker and the drying process is longer, which causes the mould weakening, and thus lowering its structural strength.

The liquid ceramic slurries containing 68% of solid phase (Ludox AM and SK) is considered the optimal one in respects of reological properties. Diagrams 11 and 12 present the influence of the solid phase fraction (Al_2O_3), on the thickness of deposited layers. Measurements were also performed for slurries containing 67, 68, 69% of the solid phase, based on the Ludox AM and SK binders.

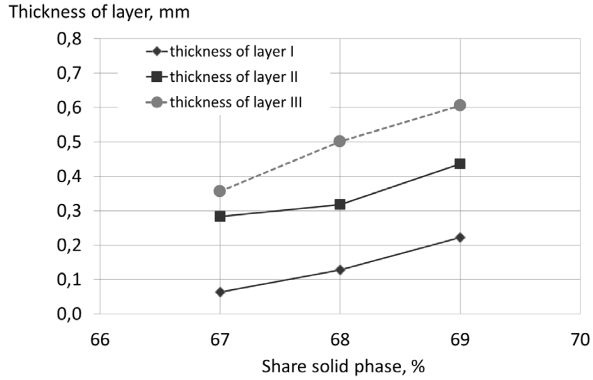

Fig. 11. Influence of the Al_2O_3 fraction in the ceramic mixture made on the base of the Ludox AM binder, on the thickness of layers formed on wax models

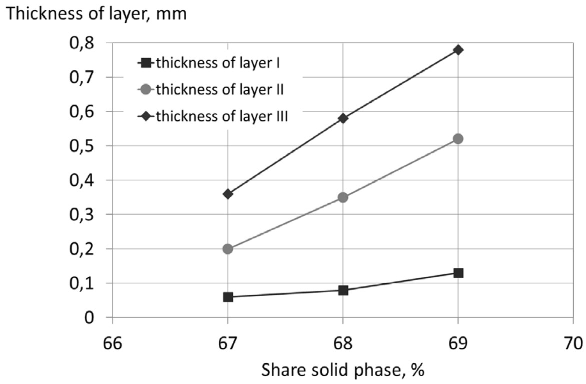

Fig. 12. Influence of the Al_2O_3 fraction in the ceramic mixture made on the base of the Ludox SK binder, on the thickness of layers formed on wax models

It results, from the performed investigations, that an increase of the solid phase content causes the thickness increase of deposited layers. This is a disadvantageous effect for the ceramic moulds production. The thicker will be the produced layers the longer will be their drying process and - in consequence - obtaining the proper strength of the mould will be difficult.

5. Conclusions

The written below conclusions are formulated on the bases of the performed investigations:

- Ultrasonic investigations allow to indicate accurately the drying time of each deposited layer of the liquid ceramic slurries.
- Ultrasonic investigations allow to state that the first layer is subjected to the highest hydration after depositing the second layer. It concerns the slurries based on the aqueous Ludox AM binder as well as SK binder.
- Deposition of the second layer causes a temporary loss of elasticity and weakens for a moment the mould structure.

- An increase of the solid phase content in the ceramic slurries causes prolongation of drying times of deposited layers. This results from the fact that the side effect of increasing the solid phase fraction in the liquid ceramic slurries is its viscosity increase and - in consequence - thickness increase of the formed ceramic mould.
- Too high slurries viscosity causes depositions of thicker and thicker layers, which makes drying more difficult and can cause cracks.

Acknowledgements

The studies have been financed within the Dean's Grant no. 15.11.170.545.

References

[1] Allendorf, A. (1960). *Using Precision Foundry Melted Models*. Warsaw: PWT. (in Polish).
[2] Haratym, R., Biernacki, R., Myszka, D. (2008). *Ecological investment casting in ceramic dies*. Warsaw: Warsaw University of Technology, Publishing House. (in Polish).
[3] Haratym, R. (2005). Anisotropy of properties of ceramic molds in aspect of accuracy of castings made in the counter-pressure process. *Archives of Foundry*. 15, 115-120. DOI: 10.151515.
[4] Matysiak, H., Haratym, R. & Kurzydłowski, K.J. (2008). Evaluation of properties of multilayer ceramic moulds in the aspect of environmental protection. *Archives of Foundry Engineering*. 8(2), 27-30.
[5] Zych, J. & Kolczyk, J. (2013). Kinetics of hardening and drying of ceramic moulds with the new generation binder – colloidal silica. *Archives of Foundry Engineering*. 13(4), 112-116. DOI: 10.2478/afe.
[6] Filipczyński, L., Pawłowski, Z., Wehr, J. (1963). *Ultrasonic testing methods of materials*. Warsaw: WNT.
[7] Show, A.D. (2002). Condition monitoring using ultrasound. Foundry Trade Journal 11, 24.
[8] Mikulczyński, T., Bogdanowicz, J., Dworzak Ł. & Ganczarek M. (2006). Valuation of active binder content in moulding sand by means of ultrasonic method. *Archives of Foundry*. 6(20), 59-64. DOI: 10.151515. (in Polish).
[9] Mikulczyński, T., Ganczarek, M., Ciskowski, S. & Dworzak, Ł. (2006). New method for determining the content of active binders in the molding mass. *Archives of Foundry*. 10-11, 574-577. DOI: 10.151515.
[10] Zych, J. (2006). PL 192202 B1. Cracow. Patent Office of the Republic of Poland.
[11] Zych, J. (2003). Investigations by the nondestructive method of the setting process of the quick setting moulding sands purged with gases. *Archives of Foundry*. 3(10), 297-306. DOI: 10.151515.
[12] Zych, J., Matysiak, H. & Michalski, J. (2009). Kinetics of hardening of ceramic layers applied in the investment patterns method. *Archives of Foundry Engineering*. 9(2), 151-154. DOI: 10.2478/afe.