

# Kinetics of hardening of ceramic layers applied in the investment patterns method

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## Abstract

The work presents results of investigations of a hardening rate of ceramic moulds applied in investment patterns method. During the studies, the original method based on the ultrasonic technique, and a novel testing routine were used. The course of hardening of single ceramic layers, and subsequently applied layers was determined. The investigations concerned ceramic mixtures with a binder based on water colloidal silica and  $Al_2O_3$  matrix. The effect of ceramic layer thickness on its hardening at temperature;  $T = 23-24^\circ C$  and, air relative humidity;  $RH = 45-55\%$  was determined.

**Key words:** innovative materials and casting production technologies, investment compound, shell moulds, ultrasounds

## 1. Introduction

Manufacturing of ceramic moulds for the precise casting (investment pattern method) consist in cyclic dipping of a wax pattern in liquid ceramic moulding sand, its powdering with coarse-grained ceramic powders and drying of such produced layer until a suitable thickness and mechanical strength of a layered ceramic mould is achieved. Application of successive layers should be carried on after sufficient drying of the previous ones. Application of layers before drying of the previous ones may lead to extensive weakening of whole, multilayered construction of the mould. Moreover, drying of thicker and thicker layers extensively elongates, which makes the process technology less effective. Determination of the layers drying time may be, with certain approximation, base on weight loss measurements (evaluation of evaporation of volatile compounds of ceramic moulding sand). However, this

method is not accurate, and it does not contain information about increase in strength of the drying ceramic layer.

Recently, a new method of investigations of hardening process of ceramic materials containing a binder was developed [ 1-5 ]. The method is based on ultrasonic technique [3]. The essence of the ultrasonic investigations which make possible to monitor the kinetics of hardening of the ceramic layers is on-line measurements of the ability of a given (hardening) medium to propagate ultrasonic wave. Hardening of the ceramic layer causes increase of the propagating wave velocity. The increase of the wave velocity is a measure of degree of the ceramic layer hardening; it is an indicator of increase of elasticity and strength of the ceramic layer.

This work presents results of investigations of kinetics of hardening of ceramic layers based on binders containing colloidal silica. Water binder contains colloidal particles of nanometric size ( $10^{-9}$  m). The silica particles suspended in water are surrounded by an electrical double

layer which prevents them from agglomeration i.e., they form a stable suspension (sol). During water removal (drying) silica particles connect one with another and form agglomerates, coalescent which lowers their specific surface, and thus free surface energy, which is favourable from a thermodynamic point of view.

Silica is lyophobic (i.e., hydrophilic) and it does not gel until a solvent is removed, which enables particles to connect one to another. The particles form an irreversible gel due to van der Waals forces. Transition of the binder from sol to gel state alters its ability of ultrasonic wave propagation. Measurement of the ultrasonic wave velocity makes possible to estimate of degree of drying and hardening process of the ceramic layer.

## 2. Methods

A multilayer ceramic shell prepared for the ultrasonic investigations was applied on a 'model' made of rubber (Gumosil) as presented in Fig.1. In order to force ultrasonic wave propagation through the hardened ceramic layer, and to create conditions of the ultrasonic wave measurement only in this layer, and not in a core, the core was made from a material which strongly dampens the waves. Although, the wave was transmitted by both media, the receiver head records only a wave signal passing through the ceramic layer. The wave velocity is determined on the basis of time of passing of the ultrasonic wave through the layer of a given length, Fig.1. The time is recorded by a computer at defined time intervals e.g., after 30 s.

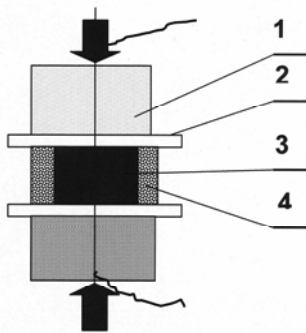


Fig.1. Scheme of ultrasonic investigations of hardening of thin, ceramic layers, which are uncovered (i.e., dried) from one side; 1 – ultrasonic heads, 2 – plates coupling a sample with the heads, 3 – core of the testing sample, 4 – ceramic layer (investment compound)

The actual appearance of the samples used in the investigations of the ceramic layer hardening kinetics is presented in Fig.2. On the left there is a sample before

coating with the ceramic layer, and on the right is a sample after covering with the few ceramic layers.

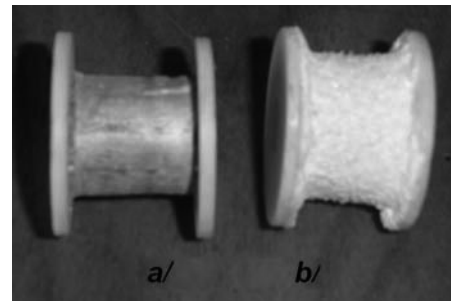


Fig.2. Appearance of a sample for the ultrasonic investigations of hardening of thin, ceramic layers, which are uncovered (i.e., dried) from one side; *a* - before, and *b* - after covering with the ceramic layer

Samples covered with the ceramic layer were inserted into the testing chamber shown in Fig.3. During the layer hardening (drying), an ultrasonic wave passed through the sample in short time intervals, and its velocity was recorded by the testing system i.e., an ultrasonic tester connected to PC. In the testing chamber constant temperature and air humidity were maintained. Ceramic moulding sands based on water colloidal silica as a binder were dried in the following conditions; temperature – 23-24°C, relative humidity 45-55%, which are similar to conditions in which industrial moulds are prepared.

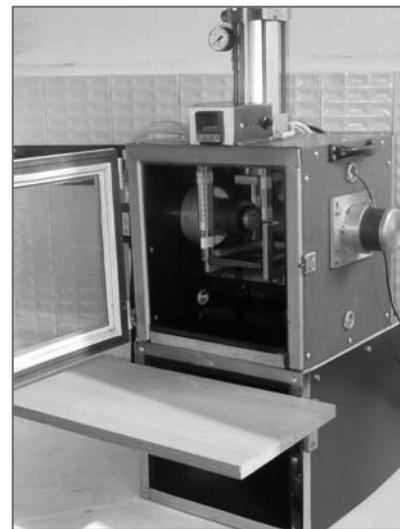


Fig.3. Testing chamber for the ultrasonic investigations of hardening of moulding and ceramic sands [1,2].

### 3. Results

#### 3.1 The kinetics of hardening of the first layer

Investigations of hardening of a first layer made of investment compound prepared in a laboratory conditions (i.e., not in industrial) were carried out. The investment compound was prepared from the following components: water binder – colloidal silicas, Ludox SK or Ludox AM; and matrix –  $Al_2O_3$  powder. The  $Al_2O_3$  powder was used to prepare both, the investment compound (coarse-grained fraction), and as a matrix material used for powdering subsequent layers during formation of the ceramic mould (shell). After application of a single layer of the ceramic sand and after dripping of its excess, the surface of the mould was powdered with a matrix powder i.e.,  $Al_2O_3$  with a particle size fraction corresponding to a given layer number (i.e., its position in the mould). First two layers were powdered with fine-grained fraction of  $Al_2O_3$ , while the others with the coarse-grained one.

In a description of the hardening kinetics of ceramic moulds based on the ultrasonic method, velocity of a longitudinal wave in the hardening layer was used. The wave velocity follows the increase in elasticity and strength of the hardening ceramic sand [2].

The course of the hardening, depicted in Fig 4, can be divided into three stages;

I – a stage in which changes of the wave velocity are almost “invisible”, and are not recorded during the test (the ceramic layer does not gain strength),

II – a stage in which changes of the wave velocity are significant and very fast (the investment compound strengthens),

III - a stage in which increase of the wave velocity is much lower, but still noticeable (the investment compound still strengthens, but very slowly).

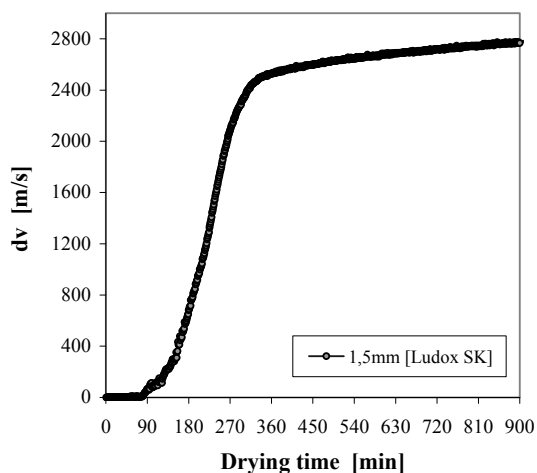


Fig. 4. Increase of the wave velocity during hardening of the investment compound consisting of; a colloidal binder Ludox SK and  $Al_2O_3$  matrix;  $T=23-24^{\circ}C$ ,  $RH_{Air} = 45-55\%$

Velocity vs. time curve presented in Fig.4 enables to determine a duration time of the first and second period. Differentiation of the curve ( $dv/dt$ ) makes possible to follow the ceramic sand hardening kinetics more precisely, to determine instantaneous rates of these changes, and to determine start and termination of the particular stages, Fig.5. For the process technology of preparation of the multilayered ceramic mould it is important to determine a duration time of the stage I, and duration of the first two stages. In the analysed example of hardening of ceramic sand with Ludox SK binder length of these stages is 90 and c.a. 360 min respectively. Termination of the second stage is a most favourable moment for application of a successive layer of investment compound. Further drying of this mould layer only elongates time of the technological process, and does not influence strength of the mould (it is unnecessary).

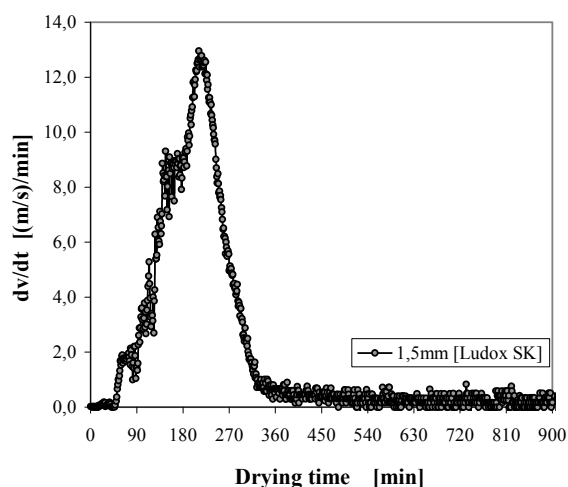


Fig. 5. Rate of hardening of a layer consisting of; Ludox SK binder, and  $Al_2O_3$  matrix;  $T=23-24^{\circ}C$ ,  $RH_{Air} = 45-55\%$

#### 3.2 Effect of the ceramic layer thickness

The ceramic layers applied on a wax model may have a different thickness, which depends on rheological properties of a binder, mainly on its dynamic viscosity. In the industrial conditions, the viscosity is repeatedly controlled, and kept in a narrow range for each system. It makes an opportunity to produce layers with identical thickness. However, in the actual industrial conditions, in case of complex patterns a phenomenon of formation of beads in corners and pits of the pattern surface is observed. For that reason, it is important to determine the hardening kinetics of ceramic sand in layers with a technological thickness i.e., slightly above 1 mm. It is also important to learn the hardening kinetics in distinctively thicker layers (c.a. 3.0 mm). Results of the hardening kinetics studies in layers with 1.5mm and 3.2 mm thickness are presented in Fig.6. Increase of the layer thickness from 1.5 mm to 3.2 mm caused elongation of the stage I from c.a. 90 min to c.a. 260 min, and elongation of duration of the first two stages from c.a. 500 min to approximately 780 min. It means that, when keeping the layer thickness at c.a. 1.5 mm the next layer can be applied after c.a. 8h, and when the thicker layer is formed (c.a. 3.2mm) the application should be performed after

c.a. 13h. It is a significant difference in duration time of subsequent technological operations

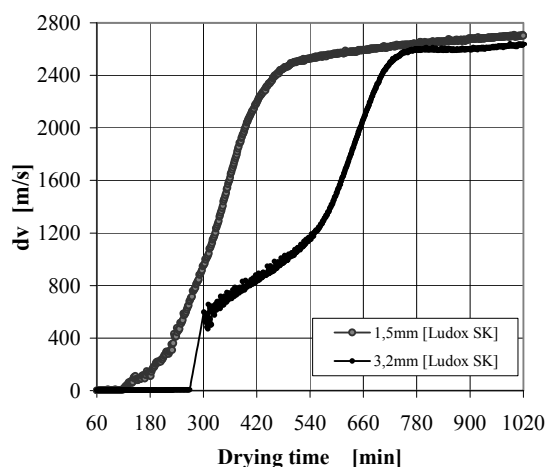


Fig. 6. Increase of ultrasonic wave velocity during hardening of the ceramic layers of 1.5mm and 3.2mm thickness. The layers consisting of; Ludox SK binder, and  $Al_2O_3$  matrix;  $T=23-24^{\circ}C$ ,  $RH_{Air} = 45-55\%$

### 3.3. The setting kinetics in successively applied layers

Fabrication of a ceramic mould on a wax pattern requires application of few, usually 8 to 10 layers. The application of successive layers with the use of colloidal silica causes temporary changes in strength of previously formed and, hardened by drying layers. The liquid binder penetrates pores of the previous layer and weakens it. The presented ultrasonic method, due to its non-destructive character, enables to monitor the above described phenomenon in multilayered ceramic moulds.

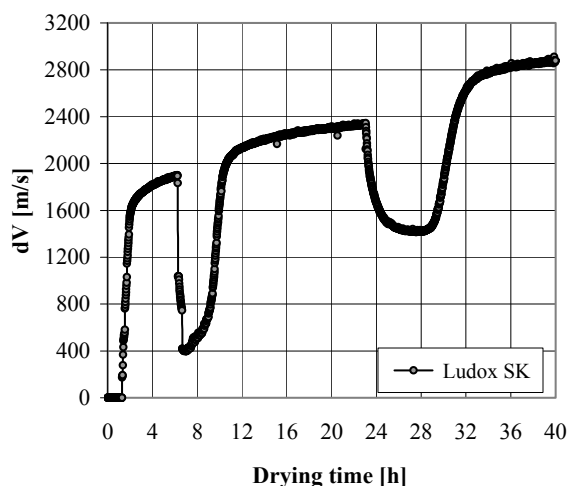


Fig. 7. Course of hardening in subsequently applied layers of a ceramic mould. The layers consisting of; Ludox SK binder, and  $Al_2O_3$  matrix;  $T=23-24^{\circ}C$ ,  $RH_{Air} = 45-55\%$

Fig.7 presents a hardening course recorded during drying of three successively applied layers. The application of the second layer “moves back” the hardening (strengthening) of the dried first layer. This weakening is caused by re-hydration of the ceramic sand forming a thin (c.a. 1.5 mm) layer. In the initial stage of drying, the ceramic mould consisting of two layers has very low strength. Application of a third and subsequent layers does not lead to such extensive hydration of preceding layers of the mould and their distinctive weakening. The recorded course of the hardening kinetics of subsequent layers makes possible to determine a duration time of particular stages of drying, which can be used for elaboration of the whole technological process of a ceramic multilayered mould fabrication. This is the target objective of the presented investigations of the ceramic layers hardening kinetics

### Summary

The work presents a novel, ultrasonic method of investigations of the hardening kinetics of ceramic moulds applied in the investment pattern method. A non-destructive character of the ultrasonic investigations performed on-line enables to monitor a hardening process of any ceramic layers, which are uncovered from one side. The research serve to determine the hardening times of subsequent layers, which can be used to optimise the process engineering of production of ceramic moulds used in the investment patterns method.

### Acknowledgments

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