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# Dilatometric studies of plaster sandmix in raw and heat treated state

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## Abstract

Results of dilatometric studies of bounded plaster sandmix applied in precision pressure below atmospheric casting, are presented in this paper. Sandmix composed of half-hydrate  $\alpha$ -CaSO<sub>4</sub>·2H<sub>2</sub>O with different parts of silica SiO<sub>2</sub> was a subject of investigations. Silica is a factor weakening the influence of phase transformations on total distortion of the mould during heating and thus influences the accuracy of prepared cast. Experimental moulders of dimensions  $\emptyset$ 7x35 made of plaster sandmix with silica fraction equal 30; 40; 50; 60 and 70% were used during studies. Sandmix was tested in raw state and after heat treatment changing the  $\alpha$ -half hydrate into anhydrite II (CaSO<sub>4</sub>). It was demonstrated that addition of the silica at level about 50% influences most advantageously on dimension changes of heated sandmix by lowering dilatations 3 times in comparison with sandmix composed of pure  $\alpha$ -half hydrate. The transformation of plaster structure into anhydrite II is also important - the shrinkability phase disappears and expansion similar to linear-like appears. It was determined that it is possible to obtain sandmix of small, stabile distortion on the way of appropriate selection of components and heat treatment parameters what improves dimensional and shape accuracy limits of the cast and significantly limits internal stresses in the mould eliminating risk of its cracking.

Keywords: Innovative foundry technologies and materials; Precision casting; Plaster mould; Dilatation

## **1. Introduction**

Technology of casting in plaster moulds is quite complicated and requires very accurate adherence to technological standards. It is also time consuming and the heat treatment of the mould is quite energy consuming. The reasons for those are first of all phase transformations in heated plaster. During heating of bounded dihydrate  $\alpha - CaSO_4 \cdot 2H_2O$  it transforms first into halfhydrate  $\alpha - CaSO_4 \cdot 0.5H_2O$  and next into unstable soluble anhydrite III and in turn into stable, insoluble anhydrite II. Those transformations proceed with changing of the degree of hydration and rebuilding of crystal lattice from monoclinic system into orthorhombic one, characteristic for anhydrite II. Those changing are accompanied with changes in densities: dihydrate ~2310 kg/m<sup>3</sup>, half-hydrate ~2755 kg/m<sup>3</sup>, soluble anhydrite III ~2484  $kg/m^3$ , insoluble anhydrite II ~2955 kg/m<sup>3</sup>, respectively. These processes cause significant, violent changes of mould material, what in result produces stresses, which, at too fast heating can cause distortions and even cracking of the mould.

Independently on given above problems this technology especially with use of pressure below atmospheric, allows obtain casts of high dimensional accuracy, very good shape representation, low roughness and clean surface. Castings of very complicated shape and thin walls, even of thickness below 1mm can be obtained by this method [2].

These advantages make this method useful in art casting, jewellery, prosthodontia and low series industrial precision casting of non-ferrous alloys as an alternative for chill casting.

Former studies revealed [3] that the addition most advantageously influencing dimensional changes of plaster bounded sand is a silica due to specific character of phase transformation proceeding in it during heating [4].

It is characteristic, that negative properties of the plaster change significantly after high-temperature  $(700 \div 750^{\circ}C)$  heat treatment. Anhydrite II obtained then is in fact ",dead' material characterized, as distinct from plaster, by not abruptly changing shrinkage but lower linear expansion [5].

The aim of our studies was to determine the characteristics of dimension variations of plaster with different fraction of silica dependently on heating temperature, for raw sandmix and after heat treatment (after transformation into anhydrite II).

## 2. Methodology and scope of studies

#### 2.1. Scope of studies

Studies on dimensional variations of heated plaster sandmix were carried out with use of experimental moulders made of di-hydrate plaster ( $\alpha$ -CaSO<sub>4</sub>·2H<sub>2</sub>O) with following fractions of silica: 30, 40, 50, 60 and 70 %.

Moulders of the same compositions heat treated, after transformation into anhydrite II (CaSO<sub>4</sub>), were tested for comparison.

Upper limit of the temperature was set to  $850^{\circ}$ C, because above this value an noticeable process of plaster decomposition into burnt lime and sulfur trioxide [1]:

CaSO<sub>4</sub>+Q=CaO+SO<sub>3</sub>.

Former derivatographic studies [6] pointed, however, that this temperature could be too high. Preliminary studies of the pure plaster showed that above 750°C significantly increase the shrinkage of the material. [1, 6]. Thus, the upper limit of the temperature was set at 730°C. Lower limit was set at 30°C for the sake of dilatometer capacity. Heating and cooling times of the moulders were assumed on the base of preliminary investigations [5, 6].

### 2.2. Methodology

Test were made with use of DA-2 Z-TECH dilatometer. Typical moulders, for this apparatus were used of dimensions  $\emptyset$ 7x35 mm.

Principles of dilatometric test were established on the base of preliminary studies:

- 1. Heating up the moulder during 6000 s in the range of temperatures  $30 \div 850^{\circ}$ C, at heat up rate v=0,14 K/s (pure plaster) and in the range  $30 \div 730^{\circ}$ C at heat up rate v=0,12 K/s (silica composition).
- 2. Holding 15 s in maximum temperature.
- 3. Cooling down of the moulder during 1200 s in the temperature range  $850\div30^{\circ}$ C, at cooling rate  $\nu$ =0,68 K/s (pure plaster) and in the range  $730\div30^{\circ}$ C at cooling rate  $\nu$ =0,58 K/s (silica composition ).

Experimental moulders were stored in tightly closed containers, additionally were dried for 2 hours at 40°C before testing and cooled down in exsiccator to ambient temperature.

#### 2.3. Materials tested

Following materials were tested:

a) autoclaved plaster  $\alpha$  Hartform HF1 delivered by Formula of following properties:

- water-plaster ratio for liquefaction Ø120 mm – WG=0,34		
- start of binding		$-t_{wp} = 10,5 \text{ min}$
- end of binding		$t_{wk} = 15,5 \text{ min}$
- bending strength after 24 hrs		$-R_{g}^{u} = 10,55$ MPa.
b) silica $SiO_2$ of following properties:		
- chemical composition	SiO <sub>2</sub>	- 99,15%
	$Fe_2O_3$	- 0,09%
	$Al_2O_3$	- 0,39%
	CaO+MgO	- 0,25%
	Na <sub>2</sub> O+K <sub>2</sub> O	- 0,08%
- granularity	sieve 0,075	- 8,42%
	bottom	- 91.58%
- sintering temperature	$t_{s} = 1400^{\circ}C$	
- density	$\rho = 2610 \text{ kg/m}^3$	

c) distilled water

d) monosodium citrate (binding time regulator)

Moulders for tests were prepared as follows:

- mixing of composition in homogenizer LH during 1 h
- preparation of liquid plaster mix (impregnation -30 s, mixing -60 s),
- cylindrical moulders preparation (six cavities mould, running 30 s, vibration 15 s),
- drying in natural drought during 48 h,
- drying in 40°C during 2 h (for raw moulders),
- heat treatment according to scheme presented in figure 1 (for anhydrite moulders).



Fig.1. The scheme of heat treatment process for moulders a) pure plaster b) composition with silica

### 3. Dilatometric studies

Results of dilatometric studies are presented in figures 2, 3, 4, 5 and 6.

# **3.1.** Pure α-half-hydrate and anhydrite investigations



Fig. 2. Relative change in s dimension vs temperature t of plaster moulder in raw state (a) and heat treated (b). Measurement in the range of temperatures 30÷850°C



Fig. 3. Relative change in s dimension vs temperature t of plaster moulder in raw state (a) and heat treated (b). Measurement in the range of temperatures 30÷750°C

### 3.2. Studies of composition with silica fraction







Fig. 5. Relative change in s dimension vs temperature t of plaster moulder with various silica SiO<sub>2</sub> fraction, heat treated



Rys. 6. Relative change in s dimension vs  $SiO_2$  fraction, in raw state (a) and after heat treatment (b) in temperature 750°C

## 4. Discussion

# **4.1.** Pure plaster and anhydrite studies (without SiO<sub>2</sub>)

Analysis of dilatometric curves presented in figure 2 points out that above temperature 750°C shrinkage of tested sandmix made of pure half-hydrate increases significantly. Process is relatively slowly at the beginning, and above 800°C becomes rapid reaching value of 2,5% at 830°C. From the view point of mould accuracy this is not acceptable. It was assumed that the upper limit of heat treatment temperature should be definitely lower, at level 730°C (fig. 3). The shrinkage of the mass decreases, in such conditions, by 50%, and shrinkage hysteresis considering cooling, by about 40%. It can be thought that this can decrease sharply the risk of mould cracking both during heating and cooling. In case of pure anhydrite expansion decreases slightly in such conditions (about 18%), but hysteresis considering cooling decreases by over 50%

#### 4.2 Studies of plaster compositions with silica

The character of dilatometric curves, presented in figure 4, of plaster sandmixes with silica differs significantly from corresponding curves for pure plaster. The reasons for that are specific properties of silica, thus non-uniform thermal expansion caused by phase transformations proceeding during its heating.

Tested silica extents quite intensively reaching extension  $s=0.9\div1.0\%$  in 573C. In this temperature except normal thermal expansion a rapid volume change appears caused by transformation of  $\beta$ -quartz into  $\alpha$ -quartz. This causes decrement of the  $\beta$ -quartz density by weakening the crystal lattice. Extension of the silica increases, in result of this process, to value about 1,4%.

Therefore the extension of plaster sandmix with silica is a result of dimensional changes of the two components and its value depends on theirs mass ratio.

The extension of plaster takes constant value in temperature about 600°C, dependent on  $SiO_2$  content. The cause of this is connected with fact, that in the range of temperatures  $600\div750$ °C both plaster and silica have the same values of shrinkage and extension.

Analysis of dilatometric curves presented in figure 4, shows that the lowest dimensional deviations (from zero position) has the mass containing  $50\div55\%$  SiO<sub>2</sub>. It can be assumed that at such composition mass will be loaded with the smallest thermal stresses.

Character of dilatometric curves, presented in figure 5, for mass containing  $SiO_2$  heat treated, so with plaster transformed into anhydrite II is different. Those mixtures, up to temperature about 570°C are characterized by only thermal expansion, at the beginning small, more intense in the range 300÷570°C.

Maximum value of thermal expansion in this temperature range is equal about 0,8% (for content  $SiO_2=30\%$ ) to about. 1,0% (for content  $SiO_2=70\%$ ). The significant influence of phase transformation of  $\beta$ -quartz into  $\alpha$  quartz becomes more and more crucial, the biggest for mixture containing  $SiO_2=70\%$  ( $s_{750^\circC}=1,25\%$ ), the lowest for mixture of content  $SiO_2=30\%$  ( $s_{750^\circC}=1,10\%$ ).

The dependence between maximum thermal expansion of raw plaster sandmix and heat treated sandmix (after transformation into anhydrite II) and content of silica, is presented in figure 6. It arises from the plot, that volume transformed into anhydrite II in temperature 750°C is practically dimension stabile independently on amount of silica content.

Investigated raw mixture shows the lowest dimensional deviation at  $SiO_2$  content about 45% .

## 5. Conclusions

On the base of carried out analysis following conclusion can be drawn:

- 1. The most advantageous properties from the view point of dimensional stability and minimizing thermal stresses has the plaster sandmix of silica content at level 50÷55%.
- Heat treated moulds should be applied in casting practice, because they are characterized by the lowest and the most stabile thermal expansion coefficients.
- Temperature of heating of the sandmix should not exceed 750°C.

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