



ARCHIVES  
of  
FOUNDRY ENGINEERING

ISSN (2299-2944)  
Volume 18  
Issue 2/2018

45 – 50

DOI: 10.24425/122501

9/2



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

# Modified Hot Distortion Test to Investigate the Effect of the Inorganic Binder on the High-Temperature Behaviour of Physically Hardened Moulding Sands

**M. Stachowicz**

Department of Foundry Engineering, Plastics and Automation, Wrocław University of Technology,  
ul. Smoluchowskiego 25, 50-372 Wrocław, Poland

Corresponding author: E-mail address: [mateusz.stachowicz@pwr.edu.pl](mailto:mateusz.stachowicz@pwr.edu.pl)

Received 15.08.2017; accepted in revised form 06.03.2018

## Abstract

This study is an attempt to determine by Hot Distortion Test (HDT) the impact of physical methods of hardening inorganic binders in the moulding sands on phenomena caused by influence of thermal energy from heating elements with a temperature of 900°C +/- 10°C. Medium silica sand-based moulding mixtures were densified and then hardened using two physical methods: microwave heating at a frequency of 2.45 GHz or classical drying at a temperature of 110°C. Sodium silicate bonded sand (SSBS) with five unmodified kinds of hydrated sodium silicates subjected to two different types of hardening method were assessed in terms of their behaviour in high temperature. Thermal behaviour by means of deformation measurement was carried out with a modified Hot Distortion Test (mHDT). Due to this advanced, but unstable by appropriate standards Hot Distortion Test gives an opportunity to measure thermoplastic deformations (L) in moulding sands in many aspects, such as time of annealing. Research carried out in this way exposed differences between inorganic binders with molar module ranging from 3.4 to 2.0. It was established that deformations under the influence of high temperature last the longest in SSBS containing binders with molar module ranging from 3.4 to 2.9. Similarly, for these types of moulding sands the method of hardening the binder is found to be essential for increasing/decreasing the rate of thermoplastic deformations during the annealing of samples. The samples of SSBS made with binders with molar module from 2.5 to 2.0 are found to be excessively susceptible to thermoplastic deformation as a result of heating them in high environmental temperature presence.

**Keywords:** Foundry, Sodium silicate bonded sand, Thermal deformation, Hot distortion test, Microwaves

## 1. Introduction

Moulding sands with hydrated sodium silicate are an example of easy to prepare and use in foundry conditions moulding sands with inorganic binders. Sodium silicate bonded sand (SSBS) [1] may be successfully cured by means of numerous physical techniques, like classical drying at a temperature ca. 100°C [2], or

warm air drying to a temperature of 60–80°C [3] or (after [4]) from 125 to 200°C. As a result, both water from the binder with colloidal silica (hydrated sodium silicate) and water on the matrix surface are removed. Another method of hardening the hydrated sodium silicates is microwave heating [1], which was proved in a number of studies, e.g.: into ester-microwave composite hardening method (EMCHM) [5] or into twice microwave heating process (TMHP)

[6], or using only microwave heating [1], which allows further partial regeneration [7] of used silicate binders. Apart from physical advantages of hardening methods, better strength and technological parameters of thus manufactured moulding and core sands should be taken into consideration, in particular their behaviour at the time when mould cavity is filled up.

In SSBS the mould erosion is less intensive that in moulding sands with organic binders [8], however the area affected by the erosion is greater (see Fig. 1).

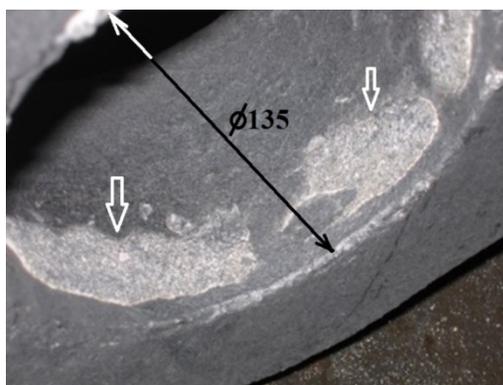


Fig. 1. Microwave hardened SSBS core erosion affected in foundry non-conformities on surface of cast-steel L120G13 elements

Therefore, this type of moulding sands (SSBS) requires protective coatings [1] in order to reduce non-conformities. Research conducted under the guidance of E. Wildhirt [9] demonstrated by Hot Distortion Test (HDT) that water-soluble protective coatings may have another negative effect on moulding sands with hydrated sodium silicate as a binder. Research on the influence of coatings should be preceded by a thorough and multidimensional analysis [10] of raw and processing moulding sands, for example by HDT-tests.

The DMA Hot-Distortion tester made for Wrocław University of Technology enable the investigation of moulding sands simulating the high environmental temperature by heated to even 990°C [11] ceramic heaters (Fig. 2). In contrast to commonly used at ambient temperature analyses of the applicability of moulding sands in the production process, HDT is useful for assessing not only strength properties, but also thermal phenomena in a given system [11,12,13]. These phenomena significantly affect the final casting shape, dimensional accuracy and smoothness. During the process of pouring liquid metal into the mould, the liquid metal intensively heats the elements of the mould and the core, either by a direct contact with those element when flowing, or indirectly when its surface rises. The decisive factor for HDT-tests is the rate of heating of the mould components. Adequate simulation of this basic test aspect is currently a major device structural and analytical problem [14].

As a result of rising temperature and mechanical stress, the moulding sand faces unfavourable and complex phenomena: polymorphous changes in the sand-base, thermal deformation, thermal and mechanical destruction as well as thermoplasticity of binders. They may lead to distortions in the mould or core structure [1, 9, 13, 15], which will further result in low quality of made casts.

This complex arrangement of thermal phenomena in the mould elements during casting and alloy solidification is the reason for using many modifications of the HDT - tests. Reports of international researchers about the thermoplastic phenomena in moulding sands indicate a significant impact of heating techniques (gas flame, ceramic or halogen heaters) of samples [12,13,16] on the results of their deformation (L). To standardize HDT they use structural and measuring enhancements such as the Hot Distortion Plus ® [13] with additional pyrometer for surface sample temperature measure and flame heating. The universal method for measuring the thermal phenomena in moulds at laboratory conditions is still not found, even in matters of the sample dimensions [11, 12, 17, 18] and the method of it heating/ annealing/ roasting.

For the purposes of research on physically hardened SSBS, taking into account the literature data and HDT device [11], the modified measurement method for thermal deformation was applied. Called by Author as “modified” HDT (mHDT) assumes the prior stabilization of the annealing temperature set at maximum (interesting) value in the DMA Hot-Distortion device heating volume. Then, entered SSBS specimen to preheated environmental volume and it's two-side heating was the basis for presented cognitive and comparative inorganic binder studies.

The sample placed in a DMA device [10] special support (Fig. 2) is heated convectively in its middle part from two sides. Unfortunately installed parallel to the sample (distance to a few mm) temperature sensor was capable of measuring ambient temperature near the sample, not the directly temperature of its surfaces (upper or lower) or temperature inside the SSBS. Under the influence of thermal energy from heating elements with a temperature of 900°C +/- 10°C, the sample due to heat transfer distorts. On the free end of the fitting, a sensor is placed which registers any changes in the sample location, i.e. deformation (L) against time and temperature.

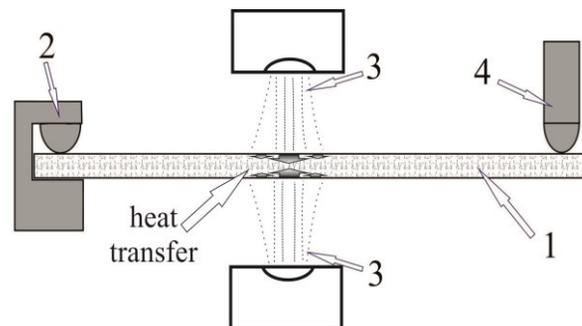


Fig. 2. Diagram of thermal deformation measuring DMA device: 1) sample of the SSBS, 2) support with a grip, 3) heat flow from ceramic heaters, 4) distortion sensor [10]

## 2. Purpose and scope of the research

Figure 3 shows a two-component system  $\text{Na}_2\text{O}-\text{SiO}_2$  and indicates a narrow range of sodium silicates with modules from 3.6 to 2.0 (typical for commercial kinds of sodium silicate).

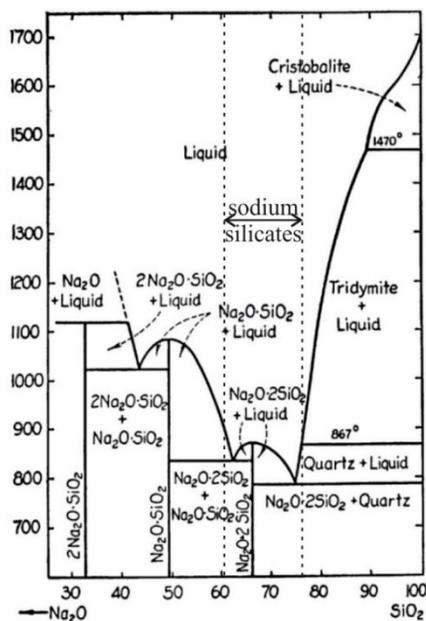


Fig. 3. Two-component system  $\text{Na}_2\text{O}-\text{SiO}_2$  with marked ranges for commercial sodium silicates [19, 20]

All the grades fall within the range with the second and third (lowest) eutectic point near  $799 \pm 3^\circ\text{C}$  [19]. Sodium silicates with low module (2.5-2.0) when heated turn into liquid state even below  $900^\circ\text{C}$ . As presented in Figure 3, an increase in  $\text{Na}_2\text{O}$  contents (higher module) in a two-component system with  $\text{SiO}_2$  results in an increase in eutectic temperature, while sodium silicate film turns into liquid state with a delay. Moreover, binders with higher module tend to be chemically bonded by  $\text{CO}_2$ , which under the influence of the environment changes the two-component system into a three-component kind:  $\text{Na}_2\text{O} - \text{SiO}_2 - \text{CO}_2$ . The behaviour of physically hardened sodium silicate moulding sands, at temperature close to the pouring the mould cavity is not fully explained. Data presented in relevant literature [19, 20] reveal that the molar module of the used binder may play an important role in moulding mixtures evaluation.

Table 1.  
Physico-chemical properties of sodium silicates used in the research of SSBS as binders of moulding sands (MS137-150)

Moulding sand type:	Binder molar module $\text{SiO}_2/\text{Na}_2\text{O}$	Oxide content ( $\text{SiO}_2 + \text{Na}_2\text{O}$ ) %	Density (20 $^\circ\text{C}$ ) $\text{g}/\text{cm}^3$	Dynamic viscosity min. (P)
MS137	3.4	36.3	1.37	1
MS140	3.0	39.4	1.42	1
MS149	2.9	44.3	1.51	7
MS145	2.5	41.5	1.47	1
MS150	2.0	43.5	1.52	1

The moulding sand used for the purposes of this study was made of dried medium quartz sand with the size of grain 1K 0.20/0.315/0.16 (according to norm PN-85/H-11001), from a

Polish mine 'Grudzeń Las' and five unmodified grades of hydrated sodium silicate produced in Chemical Plant 'Rudniki' S.A. (Table 1). Each time 1 kg of moulding sand (MS) was prepared by means of a planetary mixer: to 100 wt.% of sand-base 0.5 wt.% of water was added [2]; next, after 60 s of mixing 1.5 wt.% of selected binder was added and the whole was mixed for another 180 s.

The SSBS samples to be investigated in terms of distortions in higher temperature, with the dimensions of  $120 \times 25 \times 6$  mm ( $l \times b \times h$ ), were compacted five times by means of the laboratory rammer LU-1. The samples thus moulded were then hardened in the chamber of a microwave oven (marked as MW) manufactured by Plazmatronika, of a capacity of  $32 \text{ dm}^3$ , equipped with one magnetron and the load rotation feature. Process of SSBS microwave hardening was carried out with electromagnetic waves of a frequency of 2.45 GHz and power of 1000 W. The moulded samples were also subjected to another method of physical hardening, i.e. classical drying (marked as CD). Process of SSBS classical drying was carried out in a chamber of a laboratory dryer SL 53 TOP+ of a capacity of  $56 \text{ dm}^3$ , equipped with a fan, which made the air heated to  $110 \pm 0.1^\circ\text{C}$  circulate. For the purposes of the 30-minute drying process, an unsealed ventilation pipe was used, which enabled the escape of humidity from the drying chamber. For each variant of hardening/drying the set time ensured stabilisation of weight samples.

The analyses started from sample stabilisation to the nearest of 0.01 g by means of the scales Ohaus PA4102CM/1. The density degree was expressed as the apparent density, i.e. as the ratio of the mass of each sample after hardening/drying and cooling to the volume calculated on the basis of its dimensions. For the purposes of examining high temperature phenomena in moulding and core sands, the DMA device for HDT was made by a Polish company Multiserw-Morek [11].

The assessment includes the measurements of apparent density of correct samples, and visual inspection of samples after distortion measurements.

### 3. Results

Previous SSBS studies for the sodium silicate binders with a molar module ranged from 3.4 to 2.9 [21] shown that mould compaction affect the speed of thermoplastic deformation.

Referring to research [21] the first analysed parameter was average apparent density expressed in  $\text{g}/\text{cm}^3$ . Changing this parameter in SSBS by  $0.1 \text{ g}/\text{cm}^3$  causes significant 40% change in strength parameters [22] at ambient temperature, and also affect the rate of thermoplastic deformation [21]. The weight of the samples hardened/dried and cooled for the purposes of mHDT was compared to their capacity of  $18 \text{ cm}^3$ . The results are presented in Figure 4.

Figure 4 reveals that apparent density of moulding sands was different, though the compacting method was the same. It was noticed that the density of classically dried SSBS (CD) was always lower than the density of moulding sands hardened by microwave heating (MW). The differences for moulding sands MS137 ÷ MS149 were small ( $0.01-0.02 \text{ g}/\text{cm}^3$ ), while for MS145 and MS150 they never exceeded  $0.1 \text{ g}/\text{cm}^3$ . The density parameter can then be regarded as comparable for particular moulding sands.

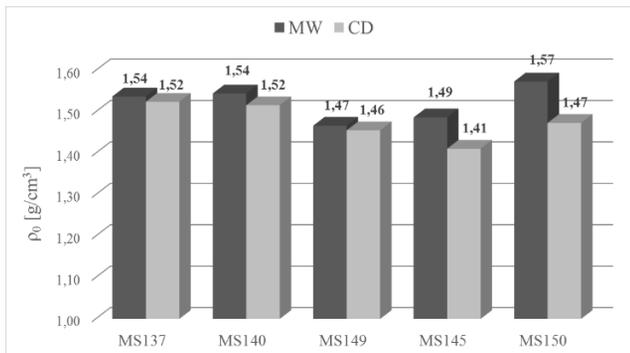


Fig. 4. Apparent density of five types SSBS: microwave hardened (MW) or classically dried (CD)

Figures 5-9 show deformation graphs received with mHDT for samples subjected to two-side heating. During stabilization of the temperature of heating elements to about 900°C +/- 10°C (see Fig. 2) the temperature sensor was outside the heating volume, like a SSBS sample. After entering the sample into a preheated volume, an increase in ambient temperature by 1°C started the registration of moulding material deformation (L).

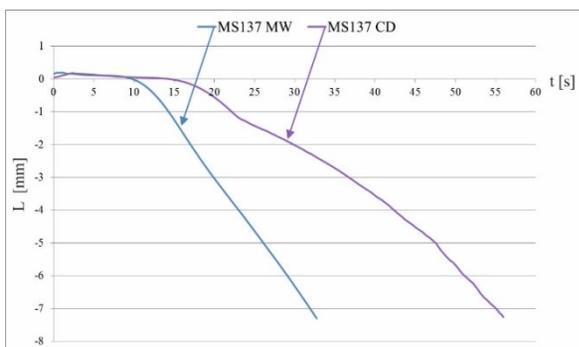


Fig. 5. SSBS sample deformation measurement results (L) in time function, for MS137 ( $\text{SiO}_2/\text{Na}_2\text{O}=3.4$ ): microwave hardened (MW) or classically dried (CD)

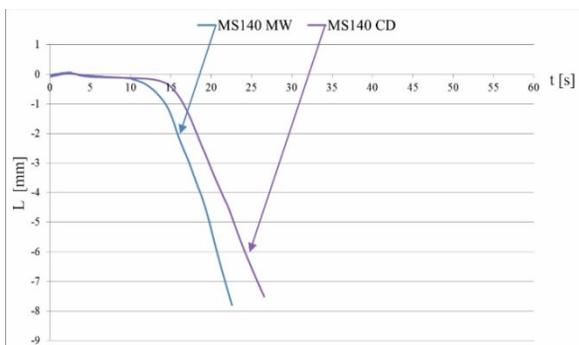


Fig. 6. SSBS sample deformation measurement results (L) in time function, for MS140 ( $\text{SiO}_2/\text{Na}_2\text{O}=3.0$ ): microwave hardened (MW) or classically dried (CD)

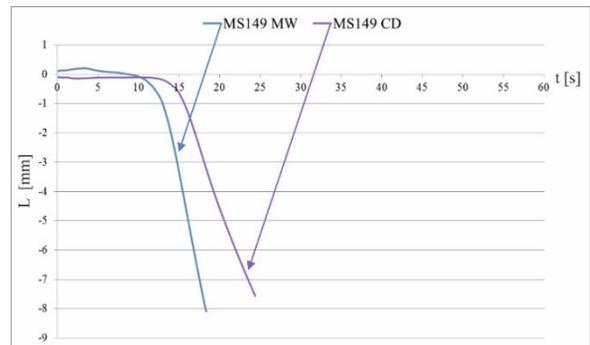


Fig. 7. SSBS sample deformation measurement results (L) in time function, for MS149 ( $\text{SiO}_2/\text{Na}_2\text{O}=2.9$ ): microwave hardened (MW) or classically dried (CD)

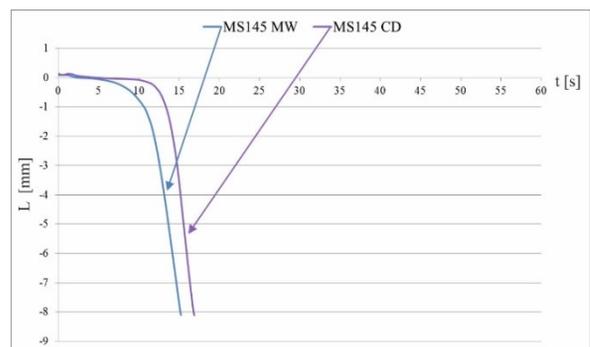


Fig. 8. SSBS sample deformation measurement results (L) in time function, for MS145 ( $\text{SiO}_2/\text{Na}_2\text{O}=2.5$ ): microwave hardened (MW) or classically dried (CD)

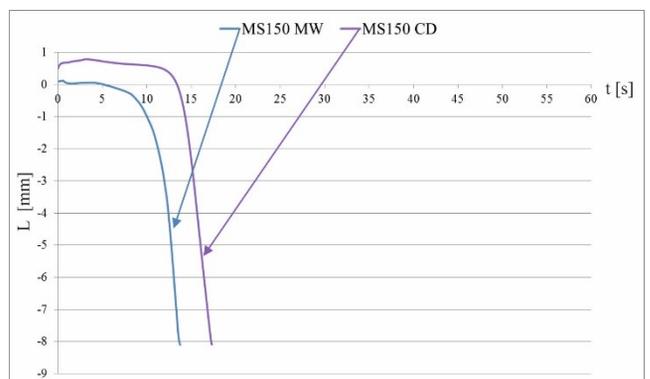


Fig. 9. SSBS sample deformation measurement results (L) in time function, for MS150 ( $\text{SiO}_2/\text{Na}_2\text{O}=2.0$ ): microwave hardened (MW) or classically dried (CD)

The analysis of all mHDT graphs revealed that physically hardened moulding sands with sodium silicate have low heat expansion, and after two-side heating, they became plasticized, without losing cohesion to the end of annealing. Each measurement was interrupted after the sample distortion (L) exceeded 8 mm. It was established that thermal deformations last the longest for moulding sand MS137 (Fig. 5) with a binder of highest molar module of 3.4 that was dried using the traditional method. Similar phenomena, though less spectacular, were detected in moulding

sands MS140 – MS150. Moulding sands of a lower apparent density (Fig. 4), hardened by classical drying (CD), reacted more slowly to the increasing ambient temperature in the vicinity of the samples. These results may contradict a previous study [21] subjected to the impact of moulding sand densification on their behaviour in higher temperature, however in this study was used only one method of SSBS physical curing (MW). The reasons behind this phenomenon cannot be put down to the issue of filling the space between grains with an insulator (air), but to differences in the process of hardening in which atmospheric CO<sub>2</sub> participates to a greater (CD) or lesser (MW) degree. CO<sub>2</sub>, taking part in the bonding reaction, changes the two-component system presented in Fig. 3 into three-component system, but the amount of used CO<sub>2</sub> remains unknown. It may be assumed that fast-setting binders with high molar module (3.4, 3.0, 2.9) will react to a greater extent with carbon dioxide than those with lower molar module, which is more visible in the plastic deformation curves (Fig. 5, 6 and 7).

Moulding sands MS145 (Fig. 8) and MS150 (Fig. 9) may be regarded on the basis of deformation curves as susceptible to excessive thermoplasticity, while the applied hardening methods had little impact on slowing the changes down.

The differences in inorganic binder grades: all registered by mHDT changes for the samples of moulding sands MS137 – MS150 are displayed in Figures 10 and 11. To supplement the comparative SSBS research Figures 10 and 11 contain additionally recorded by DMA Hot-Distortion sensor the samples environmental temperatures. The succession in results of the recorded high environment temperatures indicate the correctness in the evaluation of thermal deformation tendencies in hardened by two physical methods SSBS. As can be seen on the example of time/(temperature) measurements for MS137 - MS150 the preheated environmental volume in proposed mHDT was the basis to make a binder annealing time - deformation comparative studies.

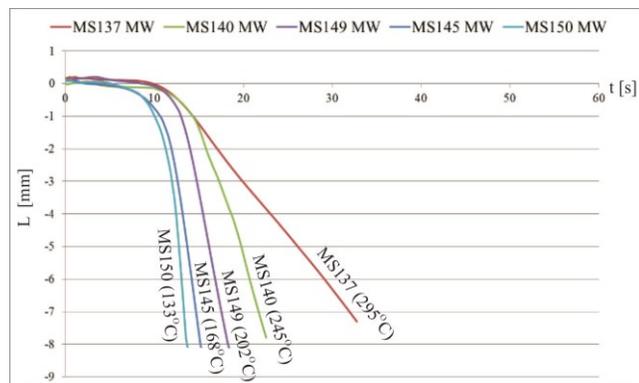


Fig. 10. Comparison of hot-distortion parameter measurements for moulding sands with five grades of sodium silicate hardened by means of microwave heating

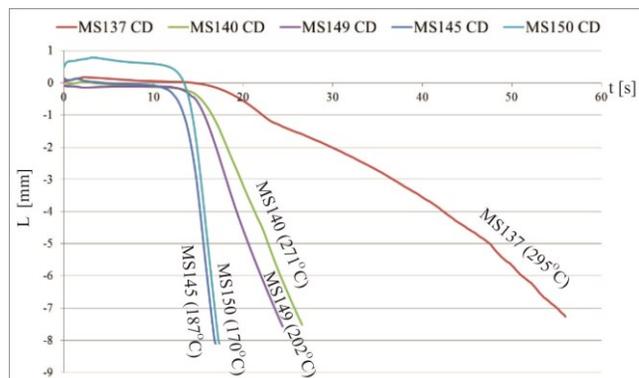


Fig. 11. Comparison of hot-distortion parameter measurements for moulding sands with five grades of sodium silicate hardened by means of classical drying

In SSBS with binders with low molar module (MS145 and MS150) the greatest deformations occurred after a shortest time notwithstanding the physical hardening method (Fig. 10 and 11). The binder in binding bridges plasticised very quickly (low eutectic temperature) and partially it turned into liquid state.

An increase in molar module over 2.9 improves the resistance to the destructive effect of alloy temperature. Then, however, worse strength and technological parameters of fresh moulding sands made with these grades of commercial inorganic binders should be taken into account.

## 4. Conclusions

The deformation measurement made with mHDT to moulding sands with five grades of sodium silicate hardened by means of microwave heating or classical drying revealed that it is the grade (molar module) of the binder that is essential, moreover:

- 1) Thermal deformations last the longest in moulding sands with binders with molar module ranging from 2.9 to 3.4 subjected to classical drying. The study also proved that it is the warm air drying, in which CO<sub>2</sub> reacts with the binder, that may be essential for slowing down the rate of deformations,
- 2) In moulding sands containing binders with molar module from 2.9 to 3.4 subjected to microwave heating, registered deformations occur faster than in the case of classical drying,
- 3) Moulding sands with binders with molar module from 2.0 to 2.5 (MS150 and MS145), the most common types of SSBS in the foundry industry, show excessive thermoplasticity that results from partial transformation to liquid state; they reveal no differences in mHDT that could result from applied hardening methods,
- 4) On the basis of mHDT deformation measurements in time function, it was found that the components of moulding sands can be selected so that proper mould and core stability will be achieved, which in turn will reduce some types of casting defects,
- 5) Research based on the HDT method, like presented in article mHDT, allows a multidimensional assessment of the inorganic binders and enables the verification of the

- previously made empirical observations of SSBS in laboratory and foundry practice,
- 6) Further research should be carried out for moulding sands with a different content of sodium silicate binders and other physical and physico-chemical methods of hardening them in order to select the most favourable environment-friendly technology of moulding and core sand.

## Acknowledgement

The research was financially supported from the grant for statutory activity No. 0402/0165/16.

## References

- [1] Liu, F.C., Fan, Z.T., Liu, X., Huang, Y. & Jiang, P. (2016). Effect of surface coating strengthening on humidity resistance of sodium silicate bonded sand cured by microwave heating. *Materials and Manufacturing Processes*. 31(12), 1639-1642.
- [2] Stachowicz, M., Granat, K. & Pałyga, L. (2016). The effect of wetting agent on the parameters of dry moulding silica sands bonded with sodium water glass. *Transactions of the Foundry Research Institute*. 56(1), 43-55.
- [3] Izdebska-Szanda, I., Kamińska, J., Angrecki, M., Palma, A. & Madej W. (2016). An innovative method for the dehydration hardening of modified inorganic binders. *Archives of Metallurgy and Materials*. 61(4), 2097-2102.
- [4] Fedoryszyn, A., Dańko, J., Dańko, R., Aslanowicz, M., Fulko, T. & Ościłowski, A. (2013). Characteristic of Core Manufacturing Process with Use of Sand, Bonded by Ecological Friendly Nonorganic Binders. *Archives of Foundry Engineering*. 13(3), 19-24.
- [5] Huaifang, W., Wenbang, G. & Jijun, L. (2014). Improve the humidity resistance of sodium silicate sands by ester-microwave composite hardening. *Metalurgija*. 53(4), 455-458.
- [6] Liu, F., Fan, Z., Liu, X., Wang, H. & He, J. (2014). Research on humidity resistance of sodium silicate sand hardened by twice microwave heating process. *Materials and Manufacturing Processes*. 29(2), 184-187.
- [7] Stachowicz, M. & Granat, K. (2016). Influence of wet activation of used inorganic binder on cyclically refreshed water glass moulding sands hardened by microwaves. *China Foundry*. 13(6), 427-432.
- [8] Zych, J. & Mocek, J. (2002). Erosion phenomenon in the moulds prepared from chemical bonded sand mould. *Archives of Foundry*. 2(3), 155-162 (in Polish).
- [9] Wildhirt, E., Jakubski, J., Sapińska, M. & Sitko, S. (2017). Impact of penetration depth of protective coating on thermal deformation of masses determined by the hot distortion parameter. *Prace Instytutu Odlewnictwa / Transactions of the Foundry Research Institute*. 56(1), 51-57.
- [10] Ramrattan, S. (2016). Non-standard tests for process control in chemically bonded sands. *China Foundry*. 13(1), 59-66.
- [11] Multiserw-Morek DMA Hot-Distortion bonded sands tester for Wrocław University of Technology– instruction manual 2016/2017.
- [12] Fox, J.T., Cannon, F.S., Brown, N.R. Huang, H. & Furness, J. (2012). Comparison of a new, green foundry binder with conventional foundry binders. *International Journal of Adhesion & Adhesives*. 43, 38-45.
- [13] Ignaszak, Z., Popielarski, P. & Strek, T. (2011). Estimation of coupled thermo-physical and thermo mechanical properties of porous ceramic material thermolabile using Hot Distortion Plus© test. *Defect and Diffusion Forum*. 312-315, 764-769.
- [14] Ignaszak, Z. (2010). Towards optimization of stress simulation in real-mold casting systems. *Archives of Foundry Engineering*. 76(4), 69-76.
- [15] Mocek, J., Zych, J. & Chojecki, A. (2004). Study of erosion phenomena in sand moulds poured with cast iron. *International Journal of Cast Metals Research*. 17(1), 47-50.
- [16] Jakubski, J. & Dobosz, S.M. (2007). The thermal deformation of core and moulding sands according to the hot distortion parameter investigations. *Archives of Metallurgy and Materials*. 52(3), 421-427.
- [17] Versatile Equipments Pvt. Ltd. Hot Distortion Tester <http://sandtesting.com/product/hot-distortion-tester/> (visit date 28.12.2017).
- [18] Morgan, D. & Fashman, E. W. (1975). The BCIRA Hot Distortion Tester for quality in production of chemically bonded sands. *AFS Transaction*. 75(91), 73-80.
- [19] Kracek, F. C. (1930). The System Sodium Oxide-Silica. *The Journal of Physical Chemistry*. 34(7), 1583-1598.
- [20] Ryś, M. (2007). *Investigation of Thermodynamic Properties of Alkali Metals in Oxide Systems Relevant to Coal Slags*. Unpublished engineering thesis, Rheinisch-Westfälischen Technischen Hochschule Aachen, Aachen, Germany.
- [21] Stachowicz, M., Paduchowicz, P. & Granat, K. (2017). Impact of density degree and grade of inorganic binder on behaviour of moulding sand at high temperature. *Journal of Casting and Materials Engineering*. 1(3), 64-69.
- [22] Zych J. (2005). Role of compaction in the casting mould technology based on moulding sands containing water-glass or chemical binding agents. *Przegląd Odlewnictwa*. 55(2), 88-97 (in Polish).