# DETERMINATION OF LENGTH CHANGES OF ALKALI ACTIVATED SLAG CONCRETE AT ELEVATED TEMPERATURES

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# Abstract:

The linear thermal expansion coefficient of two alkali-activated slag concretes produced from ground granulated blast furnace slag in the temperature range up to 1000°C is measured in the paper. Experimental results show that from the point of view of volumetric changes, both investigated alkali-activated slag concretes exhibit satisfactory response to high-temperature exposure up to 1000°C.

### Keywords:

Aluminosilicates, high temperatures, linear thermal expansion coefficient

# INTRODUCTION

Alkali-activated slag concrete was intensively studied over the past two decades as a promising alternative to Portland-cement based composites. The unique combination of high strength, considerable fire resistance and good chemical resistance which are characteristic for this type of materials attracted many investigators active in materials science, chemical engineering and civil engineering.

Mechanical properties were the most frequently investigated parameters of alkali-activated slag concrete, similarly as in the case of Portland-cement composites. They were analyzed both in room-temperature conditions and after high-temperature exposure. Other parameters such as hydric properties, chloride diffusion or thermal properties were studied only rarely. Volumetric changes of alkali-activated aluminosilicates were studied mostly in hydration stage as their drying and autogenous shrinkage was always considered a serious threat. Length changes at elevated temperatures were studied for several types of geopolymers only and in all cases remarkable thermal shrinkage (up to 20%) was observed.

In this paper, the linear thermal expansion coefficient of two alkali-activated slag concretes produced from ground granulated blast furnace slag in the temperature range up to 1000°C is measured.

#### METHOD FOR DETERMINATION OF LINEAR THERMAL EXPANSION COEFFICIENT

Thermal expansion of solid materials in high-temperature range is measured by commercially produced dilatometers mostly. Various treatments are employed, for instance the methods based on variations of electric resistance, capacitance, inductance, or the interference methods. However, the applicability of many common methods which work with small specimen dimensions may be rather limited for building materials which are mostly nonhomogeneous. Therefore, the comparative method proposed by Toman et al. [1] which was used for various building materials in the past was employed for the measurements in this paper. As some modifications of the experimental setup

were done to bring the original method up to date we will give a brief description of the method in what follows.

The measuring device for determining the linear thermal expansion of porous materials in the high temperature range is based on the application of a comparative technique (see Fig. 1). A bar sample of the studied material is put into a cylindrical, vertically oriented electric furnace. As it is technically difficult to perform length measurements directly in the furnace, a thin ceramic rod, which passes through the furnace cover, is fixed on the topside of the measured sample. The length changes can be determined outside the furnace in this way, for instance by a digital dial indicator, but on the other hand, the temperature field in the ceramic rod is very badly defined, and it is not possible to determine directly, which part of the total change of length is due to the measured sample and due to the ceramic rod.



Figure 1 Scheme of the measuring device for determination of high temperature linear thermal expansion coefficient, 1 - furnace shell (thermal insulation), 2 - furnace cover, 3 - heating coil, 4 – standard, 5 - measured sample, 6 - ceramic rods, 7 – digital dial indicator, T<sub>1-6</sub> – thermocouples

Therefore, the measurement is performed at the same time on the sample of a standard material (such as special steels where the  $\alpha(T)$  function is known) which is put into the furnace together with the studied material and is provided with an identical ceramic rod passing through the over. The change of length of the ceramic rod can be determined in this way, and consequently also the length change of the measured sample.

It should be noted that temperature field is not constant in the whole volume of the furnace due to the differences of heat loss in the heated walls and in the cover. Therefore, temperature field in the furnace is measured by thermocouples, and an average value of temperature is considered in  $\alpha$  calculations.

A practical measurement of the linear thermal expansion coefficient of a porous material with the device proposed in the previous section can be described as follows. The measured sample and the standard are put into the furnace, provided with contact ceramic rods, and the initial reading on the dial indicators is taken. Then, the electric heating regulation system is adjusted for the desired temperature  $T_i$  in the furnace, and the length changes are monitored. The data acquisition from the digital dial indicators is done on PC using specially developed software. After the steady state is achieved, i.e., no temperature changes in the furnace and no length changes are taken. The length change of the measured sample is calculated from the following formula:

$$\Delta l(T_i) = \Delta l_m(T_i) - \Delta l_s(T_i) + l_{o,s} \int_{T_o}^{T_i} \alpha_s(T) dT$$
(1)

where  $\Delta l_m$ ,  $\Delta l_s$  are the final readings of total length changes of the studied material and of the standard including the length changes of the ceramic rods, respectively,  $l_{o,s}$  is the initial length of the standard, and  $\alpha_s$  is the known linear thermal expansion coefficient of the standard. The corresponding value of thermal strain can be expressed in the form:

$$\varepsilon(T_i) = \frac{\Delta l(T_i)}{l_{o,m}} \tag{2}$$

where  $l_{o,m}$  is the initial length of the measured sample.

The measurements are then repeated with other chosen values of furnace temperature  $T_i$ , and the  $\alpha(T)$  function of the measured material is determined using the definition relation

$$\alpha = \frac{d(l/l_o)}{dT} = \frac{d\varepsilon}{dT}.$$
(3)

#### MATERIALS AND SAMPLES

Two composite materials on the basis of alkali activated slag were investigated. They differed in the type of aggregates. The first aggregate type was quartz sand (this material will be denoted as NS in what follows), the second electrical porcelain (EP). Fine-ground granulated blast furnace slag of Czech origin (Kotouč Štramberk, Ltd.) was used for sample preparation. Its chemical composition is shown in Table 1, its granulometry in Table 2.

SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	$Al_2O_3$	CaO	MgO	MnO	Cl-	Na <sub>2</sub> O	K <sub>2</sub> O	SO <sub>3</sub>
[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]	[%]
38.6	0.52	7.22	38.77	12.90	0.50	0.06	0.21	0.38	0.36

Table 1 Chemical composition of applied slag

Sieve 1	Specific surface	
0.045 mm	0.09 mm	
[%]	[%]	$[cm^2/g]$
12.4	1.9	3920

#### Table 2 Slag granulometry

As alkali activator, water glass solution  $(Na_2O . xSiO_2 . yH_2O)$  was used. It was prepared using Portil-A dried sodium silicate  $(Na_2SiO_3)$  preparative (Cognis Iberia, s.l. Spain). The quartz sand aggregates were normalized according to ČSN EN 196-1 with the granulometry given in Table 3. Electrical porcelain was provided by P-D Refractories CZ, Velké Opatovice. Its porosity was 0.3%, water absorption at saturation 0.1%, bulk density 2350 kg/m<sup>3</sup>. Chemical composition of the electrical porcelain is given in Table 4, its granulometry in Table 5.

Table 3 Sand granulometry

Sieve mesh [mm]	2	1.6	1.0	0.5	0.16	0.08
Total sieve residue (%)	0	7±5	33±5	67±5	87±5	99±1

Table 4 Chemical	composition	of electrical	porcelain
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SiO <sub>2</sub>	$Fe_2O_3$	$Al_2O_3$	CaO [%]	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>
48.6	0.8	45.4	0.3	0.2	1.0	2.9	0.7

 Table 5 Electrical porcelain granulometry

Sieve mesh [mm]		4.00	2.50	1.00	0.50	0.25	0.125	0.090	0.063	0.045
ve %]	0-1 mm fraction	-	-	0.69	45.24	70.76	89.98	93.4	98.99	99.99
tal sie idue [	1-3 mm fraction	-	4.12	78.33	99.57	99.94	99.94	99.95	98.98	100.00
To res	3-6 mm fraction	69.31	95.52	99.97	99.98	99.99	100.00	-	-	-

The composition of mixtures for sample preparation is presented in Tables 6a, b.

Table 6a Composition of the mixture with quartz sand aggregates for sample preparation

Sand [g]		Slag	Alkali-activation silicate	Water	
			[g]	admixture	[ml]
PG1	PG2	PG3		[g]	
450	450	450	450	90	190

Electr	rical porcela	in [g]	Slag	Alkali-	Water
			[g]	activation	[ml]
0-1 mm	1-3 mm	3-6 mm		silicate	
fraction	fraction	fraction		admixture	
				[g]	
450	450	450	450	90	190

Table 6b Composition of the mixture with electrical porcelain aggregates for sample preparation

The technology of sample preparation was as follows. First, the silicate preparative was mixed with water. The solution was then mixed in the homogenized slag-aggregate mixture. The final mixture was put into molds and vibrated. The specimens were demolded after 24 hours and then stored for additional 27 days in a water bath at laboratory temperature. The dimension of the specimens was  $40 \times 40 \times 100$  mm. Three specimens for each mixture were used for the measurement of linear thermal expansion coefficient.

#### **EXPERIMENTAL RESULTS AND DISCUSSION**

Fig. 2 shows the measured thermal strain of both studied materials as function of temperature up to 1000°C.



Figure 2 Thermal strain of analyzed materials as function of temperature

The  $\varepsilon(T)$  functions of both materials were increasing in the whole temperature range so that no thermal shrinkage was observed up to 1000°C, contrary to the results obtained by several investigators for geopolymers [2-4]. This difference is apparently a consequence of the presence of aggregates in the alkali-activated aluminosilicate composites studied in this paper which caused thermal expansion mismatch in the material (the gel undergoes thermal shrinkage, the aggregates expand with increasing temperature); the materials analyzed in [2-4] were pastes. This statement can be supported by the investigations in [5] where for Portland cement mortar thermal expansion was observed in the whole studied temperature range of 20 to 500°C while cement paste underwent thermal shrinkage for temperatures higher than 150°C.

The linear thermal expansion coefficients calculated using the  $\varepsilon(T)$  functions from Fig. 2 are presented in Fig. 3 which makes possible to analyze the effect of temperature increase on length changes of both studied aluminosilicates in more details. The  $\alpha(T)$  functions were increasing in the lower-temperature range, for NS up to approximately 400°C, for EP to 300°C. Then the linear thermal expansion coefficient of NS began to decrease so that at 800°C it had similar value as at room temperature, and finally in the temperature range of 800°C to 1000°C it was almost constant. On the other hand, the  $\alpha(T)$  function of EP was only slightly decreasing in the temperature range of 300°C to 700°C, and then it decreased faster so that at 1000°C it was similar as at room temperature.



Figure 3 Linear thermal expansion coefficient of analyzed materials as function of temperature

The differences between the  $\alpha(T)$  functions in Fig. 3 can be explained by the different behavior of the two materials used in the studied aluminosilicate composites as aggregates after their exposure to elevated temperatures. Electrical porcelain is characterized by low linear thermal expansion coefficient (6-8  $\cdot$  10<sup>-6</sup> K<sup>-1</sup> in average) and no sudden volumetric changes in the temperature range up to 1000°C [6, 7]. Quartz, on the other hand, is subject of displacive phase transition from  $\alpha$  (low) to  $\beta$  (high) modification at 573°C [8]. The different quartz forms differ also in their structural response to increasing temperatures. While the thermal expansion coefficient of  $\alpha$ -quartz is positive (18 · 10<sup>-6</sup> K<sup>-1</sup> in average, calculated using the data in [8] and [9]), it was found that  $\beta$ -quartz does not exhibit any significant thermal expansion from 573°C up to 1000°C [8, 9] or possibly it even undergoes thermal shrinkage [10]. Thus, the electrical porcelain aggregates had clearly better prerequisites to compensate for the thermal shrinkage of aluminosilicate gel than quartz sand, in the temperature range of 573°C to 1000°C in particular. The more convenient thermomechanical behavior of the aluminosilicate composite with electrical porcelain aggregates was probably also the main reason for its much slower decrease of strength with increasing temperature (as compared with the analogous composite with quartz sand aggregates) observed in the measurements of compressive and bending strength in [11] and [12].

A comparison of results obtained for alkali-activated aluminosilicate composites in this paper with the thermal expansion properties of other types of cementitious composites can be done in a limited extent only as very few results were published until now. In [5], the total thermal strain of cement mortar at 500°C was approximately  $9 \cdot 10^{-3}$ . This was 20% higher than for the aluminosilicate with quartz sand in this paper and almost 40% higher than for the material with electrical porcelain aggregates. The  $\alpha(T)$  functions determined up to 1000°C for cement mortar in [1] and for high performance concrete in [13] were similar in shape to the aluminosilicate material with quartz sand but 20-50% higher in the whole temperature range. As lower thermal strain generally results in lower thermal stress, thus in lower risk of failure of a building element subjected to one-sided heating, the thermomechanical behavior of the aluminosilicates studied in this paper can be characterized as mostly better than of Portland cement based composites.

# CONCLUSIONS

The experimental results obtained in this paper showed that among the two analyzed alkaliactivated aluminosilicate composites with different aggregate type, the material with electrical porcelain aggregates was found a more convenient solution from the thermomechanical point of view than a similar material with quartz sand. This was a consequence of better high-temperature volumetric stability of electrical porcelain which is characterized by low linear thermal expansion coefficient and no sudden volumetric changes in the temperature range up to 1000°C while quartz is subject of displacive phase transition from  $\alpha$  to  $\beta$  modification at 573°C and in addition, the different quartz forms differ also in their structural response to increasing temperatures.

Nevertheless, from the point of view of volumetric changes both investigated alkaliactivated aluminosilicate composites exhibited satisfactory response to high-temperature exposure up to 1000°C. The linear thermal expansion coefficient of both studied materials was in the whole temperature range of 20-1000°C significantly lower as compared to Portland-cement based composites but no thermal shrinkage was observed. This makes good prerequisites for the applications in building industry where elevated temperatures may be expected, such as walls and floors adjacent to various heat machines, pipes or chemical process vessels, envelopes or linings of special vessels such as coal gasification vessel, nuclear safety related structures in nuclear power plants, tunnel or shaft walls and fire-protecting linings.

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# REFERENCES

- [1] Toman J, Koudelová P, Černý R., A measuring method for the determination of linear thermal expansion of porous materials at high temperatures. High Temperatures-High Pressures, vol. 31, 1999, pp. 595-600.
- [2] Duxson P, Lukey GC, van Deventer JSJ, Thermal evolution of metakaolin geopolymers: Part 1 – Physical evolution. Journal of Non-Crystalline Solids, vol. 352, 2006, pp. 5541-5555.
- [3] Duxson P, Lukey GC, van Deventer JSJ, Physical evolution of Na-geopolymer derived from metakaolin up to 1000 °C. Journal of Materials Science, vol. 42, 2007, pp. 3044-3054.

- [4] Subaer, van Riessen A, Thermo-mechanical and microstructural characterisation of sodium-poly(sialate-siloxo) geopolymers. Journal of Materials Science, vol. 42, 2007, pp. 3117-3123.
- [5] Fu YF, Wong YL, Poon CS, Tang CA, Lin P, Experimental study of micro/macro crack development and stress–strain relations of cement-based composite materials at elevated temperatures. Cement and Concrete Research, vol. 34, 2004, pp. 789-797.
- [6] Amigo JM, Clausell JV, Esteve V, Delgado JM, Reventos MM, Ochando LE, Debaerdemaeker T, Marti F, X-ray powder diffraction phase analysis and thermomechanical properties of silica and alumina porcelains. Journal of the European Ceramic Society, vol. 24, 2004, pp. 75–81
- [7] Kamseu E, Leonelli C, Boccaccini D, Non-contact dilatometry of hard and soft porcelain compositions. Journal of Thermal Analysis and Calorimetry, vol. 88, 2007, pp. 571-576.
- [8] Carpenter MA, Salje EKH, Graeme-Barber A, Wruck B, Dove MT, Knight KS, Calibration of excess thermodynamic properties and elastic constant variations associated with the  $\alpha \rightarrow \beta$  phase transition in quartz. American Mineralogist, vol. 8, 1998, pp. 2–22.
- [9] Lakshtanov DL, Sinogeikin SV, Bass JD, High-temperature phase transitions and elasticity of silica polymorphs. Physics and Chemistry of Minerals, vol. 34, 2007, pp. 11– 22.
- [10] Huang LP, Kieffer J, Structural origin of negative thermal expansion in high-temperature silica polymorphs. Physical Review Letters, vol. 95, 2005, paper 215901.
- [11] Zuda L, Pavlík Z, Rovnaníková P, Bayer P Černý R, Properties of Alkali Activated Aluminosilicate Material after Thermal Load. International Journal of Thermophysics, vol. 27, 2006, pp.1250-1263.
- [12] Zuda L, Bayer P, Rovnaník P, Černý R, Mechanical and Hydric Properties of Alkali-Activated Aluminosilicate Composite with Electrical Porcelain Aggregates. Cement and Concrete Composites, vol. 30, 2008, pp. 266-273.
- [13] Toman J, Černý R, Thermal and Hygric Expansion of High Performance Concrete. Acta Polytechnica, vol.41, 2001, pp. 2-4.