

# NON-DESTRUCTIVE TESTING AND X-RAY DIFFRACTION ANALYSIS OF HIGH-TEMPERATURE DEGRADED CONCRETE

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## ABSTRACT

In the field of civil engineering a diagnostics acoustic non-destructive testing is widely used and in the past years it was used even in testing of high-temperature degraded concrete structures. These methods can provide specific information about the physical-mechanical state of material; however physical-chemical changes in microstructure are measurable to a limited extent. This article is focused on non-destructive and destructive testing of high-temperature degraded concrete test beams of dimensions  $0.1 \times 0.1 \times 0.4$  m fired at  $200 - 1200$  °C. The Impact-Echo method and ultrasonic velocity pulse method measurements are compared with destructive test results. Testing of measured changes in the p-m properties of reference and fired samples is then supplemented by X-ray diffraction analysis, in order to document mineralogical physical-chemical changes of tested material.

## KEYWORDS

Impact-Echo method, Concrete, Coarse aggregate, High-temperature degradation, Non-destructive testing, X-ray diffraction analysis

## INTRODUCTION

The presented paper investigates the potential application of non-destructive acoustic methods of testing as a diagnostic tool, used in the retrofitting process of thermally degraded concrete structures. Due to its composition, plain concrete has good fire-resistant properties, it does not produce smoke in elevated temperature, it is non-flammable and non-combustible. Its specific heat capacity is approximately  $1020 \text{ J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$  and coefficient of thermal conductivity is  $1.0 - 1.5 \text{ W}\cdot\text{m}^{-1}\cdot\text{k}^{-1}$  and thus behaves as a fine thermal insulant in case of fire [1]. Concrete is heterogeneous composite and each of the compounds, like aggregate and cement matrix, react differently to high temperature [2]. We can observe these changes by non-destructive testing. It is synergic effect of physical-mechanical and physical-chemical changes, affecting both macrostructure and microstructure. The aim of this paper is to compare the results of non-destructive testing with changes in mineralogical composition of high temperature degraded concrete.

## METHODOLOGY

A total of 63 test specimens of diameter  $0.1 \times 0.1 \times 0.4$  m were manufactured and divided into 7 temperature sets. First reference set was kept at temperature of  $20$  °C and the remaining 6 sets were divided in multiples from  $200$  °C  $1200$  °C. The composition of each mixture used in the investigation can be seen in Table 1. All tests specimens were kept in water container for 28 days.

Then each test specimen was pre-dried in laboratory dryer oven at 110 °C for 72 hours before being fired in a furnace. The length time used for pre-drying was chosen according to initial measurements of designed mixture, whereby the weight was monitored during drying until samples achieved a maintained weight. By this step we got rid of residual free water and prevented possible explosive spalling for sets degraded by 400 °C and higher [3]. The furnace temperature was set to rise at a rate of 5 °C/min and upon reaching maximum temperature for each set was maintained for 1 hour. Afterwards the specimens were cooled by atmospheric air to room temperature, inside the furnace.

*Tab. 1 - Mixture design\**

Compounds	Amount of each compound for 1 m <sup>3</sup> [kg]
Cement CEM I 42.5 R	345
Fine aggregate Žabčice 0/4 mm	896
Coarse aggregate Olbramovice 8/16 mm	521
Coarse aggregate Olbramovice 11/22 mm	391
Superplastizer Sica Viscocrete 2030	2.5
Mix water	173

*\*Compressive strength after 28 days at 20 °C was 75.1 MPa*

For non-destructive testing, both, Impact-Echo (IE) and ultrasonic pulse velocity method (US) were used. IE method is based on the excitation of low-frequency mechanical waves created within the test specimen upon mechanical impact. The test specimens begin to vibrate as the mechanical waves are reflected and refracted within their structure; these vibrations are then recorded using a suitable receiver [4].

The signal recorded in the time domain is then converted using the Fast Fourier transform [5] to the frequency spectrum, which can be easily analysed (Figure 1 and Figure 2).

Measurement by Impact-Echo was conducted as test specimens were placed on rubber pads and struck by a spherical hammer of total mass 25.5 g. The produced vibrations were recorded by a piezoelectric sensor MIDI 446s12. A digital oscilloscope Handyscope HS3 with sampling speed set at 0.2 MHz was used for signal processing.

Ultrasonic pulse velocity method is based on measuring time needed for and US pulse to travel through tested material. The presence of defects, cracks and inhomogeneity alter the ultrasonic signal, which then refracts and thus travels by longer path. Using this investigation, we can evaluate different types of degradation and localize defects within the tested structure. With knowledge of acoustic velocity in the tested medium, as well as density, and diameters of tested specimen, we can compute a dynamic modulus of elasticity [6]. For US measurement an Ultrasonic Pulse Analyser 58-E4900 was used.

By using these methods, we can evaluate the physical-mechanical state of structure, however these changes originate from changes of mineralogical composition and thus the non-destructive measuring is supplement by X-ray diffraction analysis. This method is based on subjecting the specimen to X-rays, which is reflected from each crystalline surface. According to Braag's equation, at the point of maximum reflection of X-rays, a peak is formed in the diffractogram [7]. Each peak is then identified using mineralogical libraries. Specimens for this

analysis were taken from the ends of test beams. The samples of aggregate and cement matrix were grinded to a fraction below the 0.063 mm. X-ray diffraction analysis was conducted at AdMaS centre in Brno.

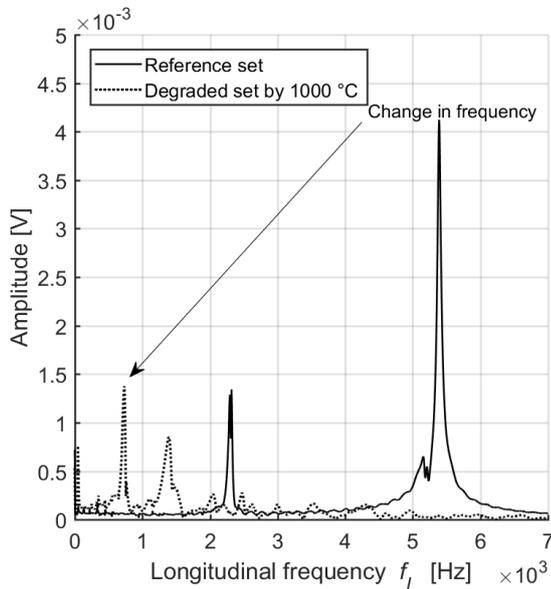


Fig. 1 – Change of dominant frequency for reference test specimen and test specimen degraded at 1200 °C.

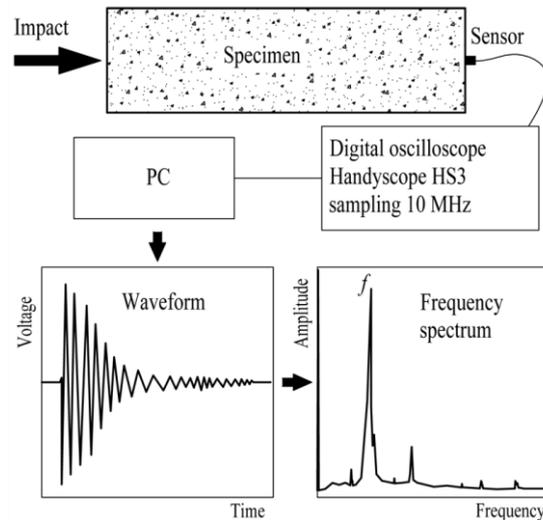


Fig. 2 – Testing setup of Impact-echo measurement.

## RESULTS

The fresh concrete prepared for manufacturing of test specimens had slump test result of S4 (60 – 70 mm) and did not show any signs of bleeding or segregation of coarse aggregate. The fresh concrete was completely compacted via a vibration table in 14 s on average. Density of concrete in its hardened state was 2380 kg·m<sup>-3</sup>. The measured change in density between non-degraded and thermally degraded state is shown in Figure 3.

The largest drop in density 11 % was observed in the temperature set of 1200 °C. The main cause of density loss is dehydration of free and chemically bounded water in aggregate and cement compounds. This smallest decrease can be observed in temperature range from 20 to 400 °C. Moreover, a decay of portlandite at 480 – 560 °C and decay of coarse and fine carbonates (CaCO<sub>3</sub> → CaO + CO<sub>2</sub>) at temperature 930 – 960 °C also accompany the loss of density [8]. From temperature 600 °C up to 1000 °C a linear trend of loss in density is observed. After the 1200 °C a steep change occurs caused by degradation of aggregate minerals and formation of ceramic bonds between melted compounds.

In terms of macroscopic changes, the thermally degraded specimens change in colour from cement grey to ochre shade of brown colour with the presence of cracks. Sets degraded at 200 °C did not show any signs of cracking, however at 400 °C, the first signs of cracking appeared. At 1000 °C the concrete became brittle and fragile to mechanic manipulation. Whole scales of dried, shrunk and cracked cement matrix were falling off, in this state the diameter of cracks reached nearly 4 mm and covered the whole body of test specimens. At 1200 °C the test specimens changed colour to brown and were recompacted by forming the ceramic bonds between the aggregate and cement matrix. The test specimens bend due to effect of transient creep [9], with total difference of middle section to edges by 2 – 5 mm.

These mechanical changes can be observed through dynamic modulus of elasticity. It is seen in Figure 4. The reference sets reached modulus of 44 GPa. From this point the designed mixture showed a decrease in measured dynamic modulus of elasticity for higher degradation temperatures. The first significant decrease in dynamic modulus of elasticity occurred between 400 and 600 °C, from 29 GPa to 14 GPa.

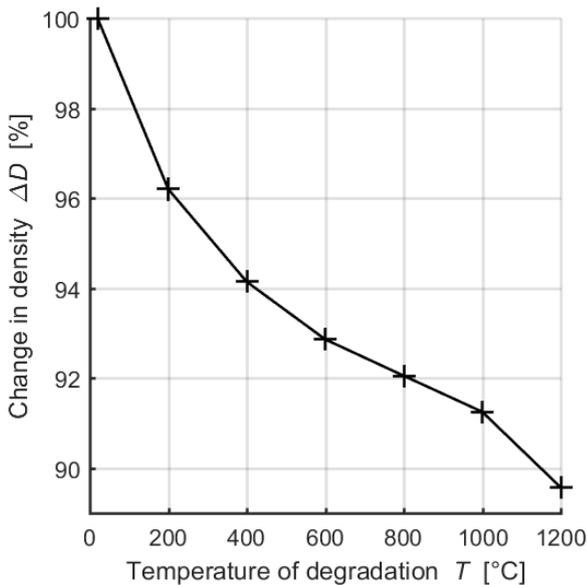


Fig. 3 – Loss in density of thermally degraded temperature sets.

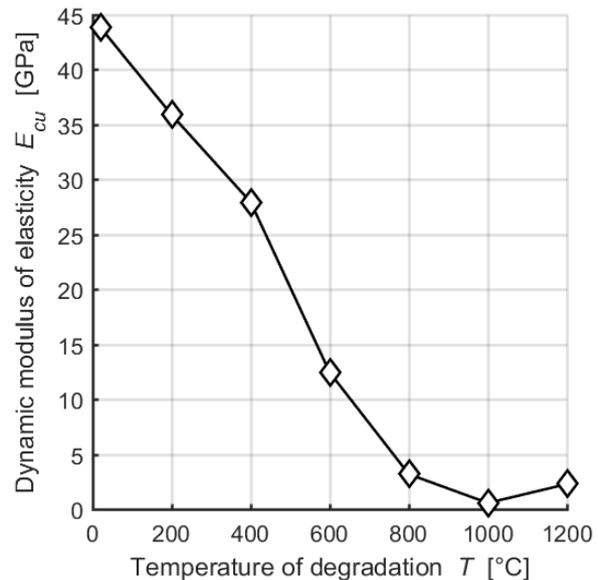


Fig. 4 – Measurement of dynamic modulus of elasticity.

From 600 °C the decrease tendency is similar and lowest measured value is 1 GPa for 1000 °C temperature set. At this temperature the test specimens had the lowest measured physical-mechanical properties.

Slightly similar results can be observed in Impact-Echo measurement shown in Figure 5. The reference sets had a resonance frequency of 5.37 kHz longitudinally and 2.30kHz transversely for the first resonance and 8.37 kHz for the second resonance frequency. The decrease in frequency was similar to change in dynamic modulus of elasticity.

The lowest frequencies were measured at 1000 °C with longitudinal frequency of 0.63 kHz and a transverse frequency of 0.32 kHz and 1.23 kHz.

Both US and IE method have confirmed the changes in physical-mechanical properties with the same trend of measured values. When we compare these results with different studies of this topic [10] the presented measured values correspond with other publications. Impact-Echo measurements have shown the ability to determine the extent of high-temperature damage.

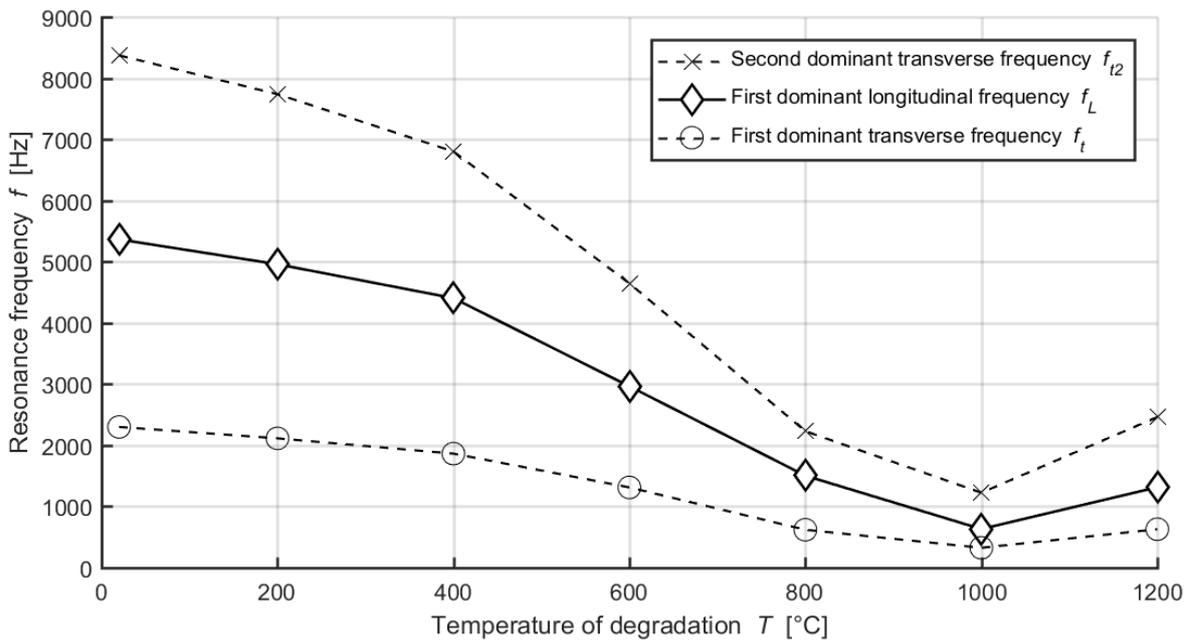


Fig. 5 – Average resonance frequencies of each temperature sets.

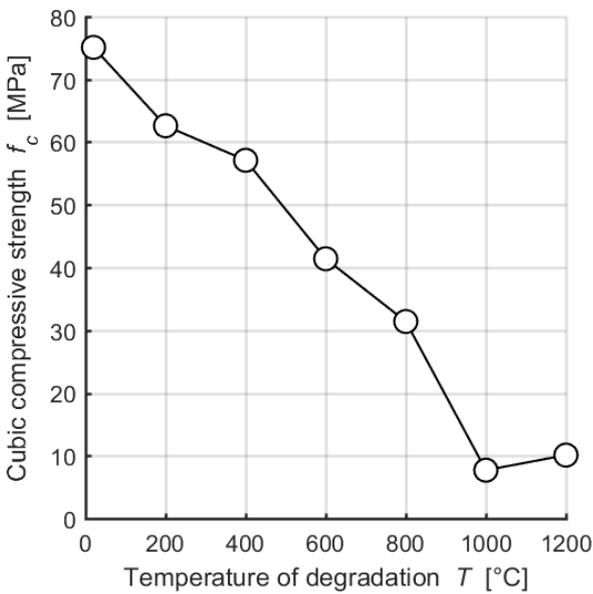


Fig. 6 – Cubic compressive strength of thermally degraded sets.

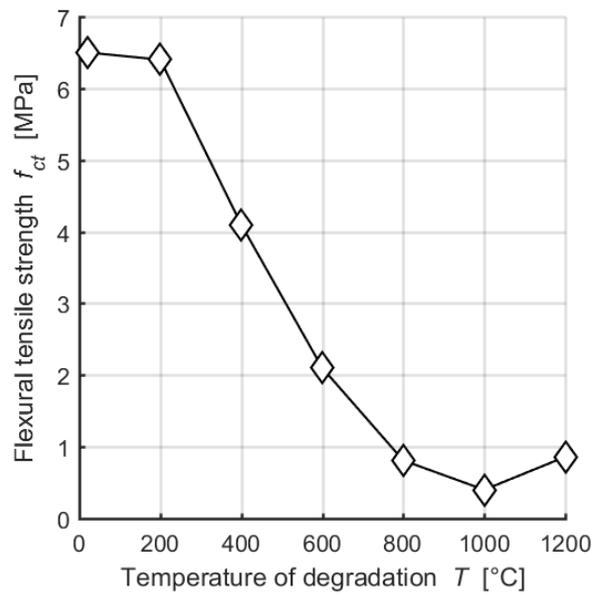


Fig. 7 – Flexural tensile strength of thermally degraded sets.

The same results can be seen in destructive tests shown in Figure 6 and Figure 7. According to destructive tests, test specimens degraded at 1200 °C had higher tensile strength than temperature sets 800 °C and 1000 °C. It seems that the tensile strength of tested material is more affected by temperature degradation than compressive strength.

Table 1 - Correlation coefficients for each measured parameter of tested material.

	$D$	$f_L$	$f_{t2}$	$f_{t1}$	$E_{cu}$	$f_c$	$f_{ct}$
Density $D$	1	0.8891	0.8858	0.8891	0.9415	0.9239	0.9168
Longitudinal $f_L$	0.8891	1	0.9985	0.9993	0.9806	0.9732	0.9707
Transverse $f_{t2}$	0.8858	0.9985	1	0.9995	0.9828	0.9612	0.9746
Transverse $f_{t1}$	0.8891	0.9993	0.9995	1	0.9809	0.9666	0.9714
Dynamic modulus $E_{cu}$	0.9415	0.9806	0.9828	0.9809	1	0.9532	0.9899
Cubic compressive strength $f_c$	0.9239	<b>0.9732</b>	<b>0.9612</b>	<b>0.9666</b>	<b>0.9532</b>	1	0.9313
Flexural tensile strength $f_{ct}$	0.9168	<b>0.9707</b>	<b>0.9746</b>	<b>0.9714</b>	<b>0.9899</b>	0.9319	1

After the non-destructive and destructive parameters were measured a table of correlation coefficients was created with results in Table 2. Apart from high values of correlation coefficients between measured frequencies we can distinguish high correlation between destructive and non-destructive tests where correlation coefficients reached values from 0.95 up to 0.97 for compressive strength and 0.97 up to 0.98 for tensile strength.

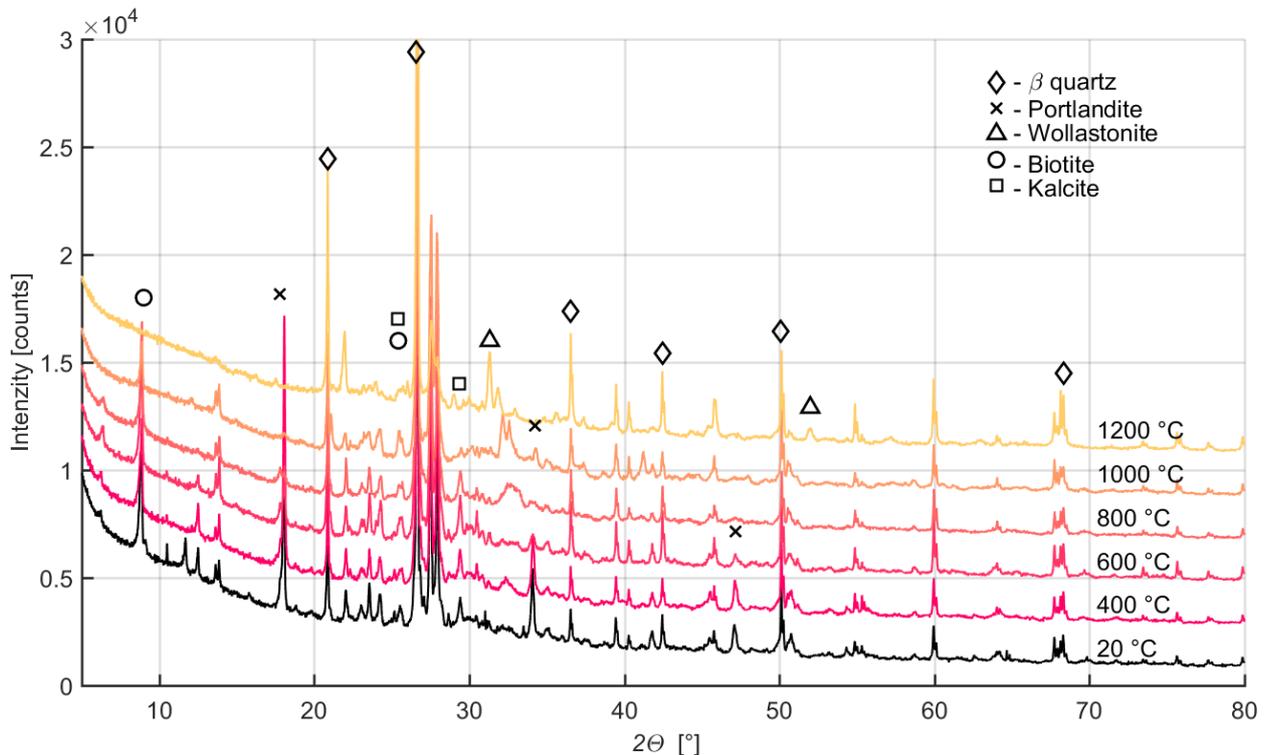


Fig. 8 – X-ray diffraction analysis of tested concrete specimens

Changes of physical-mechanical properties are based on change of microstructure and mineralogical composition of concrete specimens. For this purpose, the specimens were submitted to X-ray diffraction analysis. The measured results of reference set and temperature set from 400 to 1200 °C is shown in Figure 8. During this mineralogical analysis the presence of several minerals from both, used aggregate and cement were found. From aggregate: biotite, calcite,

kaolinite and  $\beta$ -quartz were found, while from cement: portlandite,  $\beta$ -quartz and wollastonite were found. All diffractograms were moved apart by 2000 counts for clarity. We can recognize that most changes appear between 400 and 800 °C and between 400 and 600 °C a decomposition of portlandite takes place.

Studies [11] have shown that, portlandite decomposed from 400 °C, which has been confirmed. Portlandite was recognizable with a much lower intensity at 800 °C and at 1000 °C no presence of portlandite was observed. Biotite, which is a mineral found in used aggregate decomposed between 920 and 960 °C [12], at 1200 °C biotite was not found in the diffractogram.

The hydraulic bonds between cement matrix and aggregate after 1000 °C are substituted by ceramic bonds which begin to form between calcium and quartz compounds [13]. After 1100 °C the low-melting quartz compounds start to melt and concrete shows partial sintering and forming of wollastonite  $\text{CaO}\cdot\text{SiO}_2$ . The presence of wollastonite was confirmed as it can be seen in the diffractogram from the 1200 °C set. This process is mainly responsible for recompacting of the test specimens and higher residual tensile and compressive strength.

## CONCLUSION

This paper presented an investigation designed to test the applicability of a non-destructive method as a diagnostic tool for evaluation of thermally degraded concrete test specimens. As a test method, an Impact-Echo and ultrasonic pulse velocity method was used and compared with destructive tests such as cubic compressive strength test and flexural tensile strength test. These measurements aim to compare physical-mechanical changes with physical-chemical changes in microstructure.

Non-destructive measurement shows a strong correlation with destructive tests with lowest measured values for test specimens degraded at 1000 °C. After the 1200 °C, a slight increase in residual physical-mechanical properties was observed. Partial sintering at 1200 °C of concrete test specimens was observed and was accompanied by the formation of wollastonite at 1200 °C temperature sets. Apart from the formation of wollastonite, a presence of different minerals connected with used aggregate and cement was also confirmed. A decay of portlandite at 400 - 800 °C was observed, as well as decay of biotite between 800 and 1000 °C. The Impact-Echo testing seems to be more suitable to reveal a physical-mechanical condition of thermally degraded concrete than ultrasonic pulse velocity method.

The Conducted investigation has proved the theoretical assumptions of high-temperature degradation mechanisms of concrete, on the level of microstructure, in the scope of non-destructive testing.

High coefficients of correlation between destructive and non-destructive tests indicate suitability of acoustic non-destructive testing in the field of high-temperature Cementous composites such as concrete. This ability was also suggested and proved in the abroad studies [10].

The possibility of non-destructive assessment of material is convenient approach not only in the field of building diagnostics, but also in the development of new silicate and cementous fireproof materials. The possibility of combination of results from acoustic signal, processing tools with easy and cheap resonance methods of testing and X-ray diffraction analysis can unveil specific behaviour of material which can be key in the stage of development or quality control of new materials.

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