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The Assessment of Cement Mortars after Thermal Degradation by Acoustic Non-destructive Methods

L Topolář, D Štefková and M Hoduláková

Faculty of Civil Engineering, Brno University of Technology, Veveří 331/95, Brno 602 00, Czech Republic

E-mail: Libor.Topolar@vutbr.cz

Abstract. Thanks, the terrorist attacks on the worldwide interest in the design of structures for fire greatly increased. One of the advantages of concrete over other building materials is its inherent fire-resistive properties. The concrete structural components still must be able to withstand dead and live loads without collapse even though the rise in temperature causes a decrease in the strength and modulus of elasticity for concrete and steel reinforcement. In addition, fully developed fires cause expansion of structural components and the resulting stresses and strains must be resisted. This paper reports the results of measurements by Impact-echo method and measurement by ultrasound. Both methods are based on the acoustic properties of the material which are dependent on its condition. These acoustic methods allow identifying defects and are thus suitable for monitoring the building structure condition. The results are obtained in the laboratory during the degradation of composite materials based on cement by high-temperature.

1. Introduction

Current trends in the construction industry are high demands on quality and safety of building structures then on rapid development and easy diagnosis of any structural damage. Development of automotive transportation and overall infrastructure has also consequences for the increasing number of accidents involving fire and the associated consequential damage to concrete structures in the places of fire [1].

This influences the life-time of the structure and its functionality. These problems occur not only in the transport sector but in all buildings the fire is an increasing problem that affects other behaviour of structures. An important factor in the structural design is the requirement for fire resistance of these structures [2].

In case of a fire, there is a need to verify the condition of the structure after this degradation. Objectives and outputs of generally non-destructive testing methods is the prediction of life-threatening risks and of risks associated with the use of damaged buildings. One of the advantages of these methods is that examined object after the test remains intact for further utilization.

Non-destructive methods are diagnostic methods, their main advantages are product control, and structural check in the operational phase, but also in research and development. Defectoscopy ensures reliability and safety in many industries (chemical, aerial, nuclear energy, engineering, etc.). They also provide security in the building constructions, bridges, dams and other structures. Defectoscopic methods today reveals not only defects in the structure, but also the type of defect, its dimensions and localization in the analysed object [1,3].

They are able to contribute within behaviour description of the technical structures and as already mentioned, without destructive interference of the construction. Studying these methods is the current topic in the diagnostics field.

The impact-echo method is a non-destructive acoustic technique. Its main idea is that the mechanical characteristics of a specimen affect its spectral properties. The principle of the method is based on analyzing an elastic impulse-induced mechanical wave [4]. A short-time mechanical impulse induced by a steel spherical body gives rise to a low-frequency pressure wave. Thus, the generated wave propagates through the specimen's structure being rebounded by defects located in the specimen's bulk or on its surface. The time difference between the emitted wave and the rebounded one is captured by a sensor, which shows the signal waveform. In theory, the impact resembles a delta-function containing all possible frequencies. During wave propagation, each frequency component is affected by the geometry of the sample, the material properties, defects, non-homogeneities, cracks or any other structural characteristics [5]. The frequency spectrum of the gathered signal calculated using Fourier transform (especially its dominant frequencies) give an account of the condition of the specimen.

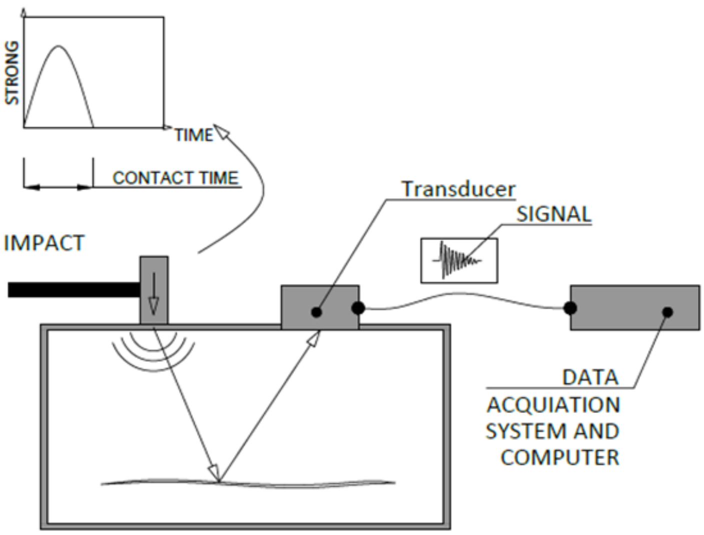

Figure 1. Principle of Impact-Echo measurement.

Ultrasound method is another method of non-destructive testing. It is based on the principle of repeated transmissions of ultrasonic pulses into a tested material (Fig. 2) and by measuring the time passage of the impulse through the material. Impulse of mechanical waves is called electroacoustic driver. This driver is placed on the surface of the examined material parallelly to the longitudinal axis of the sample [6].

For proper measurement with ultrasound pulse method, it is necessary to ensure appropriate transmission of waves from the driver (probe) into the tested material and from the tested material to the sensor. To achieve proper acoustic coupling binding agent is placed between the probe and the surface of the investigated material. Using the binding agent prevents the occurrence of an air gap between the probe and the sample and reduces the unwanted reflection of the waves and the increases in the passage from the ultrasound probe into tested body [7].

Probes with acoustic binding agent are placed at the measuring points on the opposite edges of the examined object. A thin layer of binding agent ensures good acoustic contact between the probe and the sample. Impulse vibration sensor is converted after passing through the length of the path of the material to an electrical signal, and the electronic timing circuit enables to measure the pulse transit time [8].

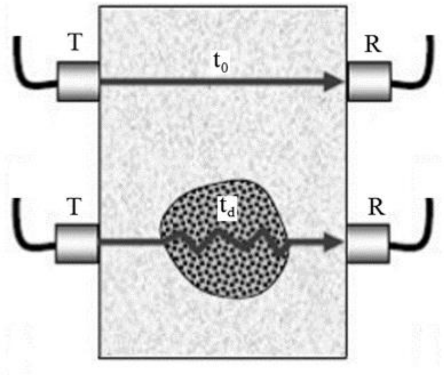

Figure 2. Principle of ultrasound measurement (T – transmitter, R – receiver) (t_0 – transit time non-degraded specimen, t_d – transit time degraded specimen)

The Non-linear Ultrasonic Spectroscopy (NUS) and is based on effect when the material has non-linear behaviour in its nature of there are so-called non-linearities inside it. Non-linearities are mainly causing by the present of flaws, defect and structure changes in it. There are few kinds of non-linearities and they have different causes and symptoms [9]. The sensitivity of NUS method is higher than wavelengths used for structure inspection and enables to reveal very small and tiny defects in initiation phase (such as developing plastic zones) - comparing to common ultrasonic inspecting non-destructive testing methods like phase array, impact-echo etc. [9,10].

2. Experimental Setup

2.1. Materials
Mortars were produced using a CEM I 42.5 R Portland cement (Českomoravský Cement - Heidelberg Cement Group) and water to cement ratio (w/c = 0.46) and quartz sand from Filtrační písky, s.r.o. for preparation mixture test mortar, in a ratio of 1 to 3. In compliance with ČSN 721200 standard. The specimens (of dimensions 40x40x160 mm) were left in the moulds for 24 hours, then cured in water for 27 days and finally air-cured for 31 days at laboratory temperature (25±2) °C a relative humidity of (53±5) %.me text.

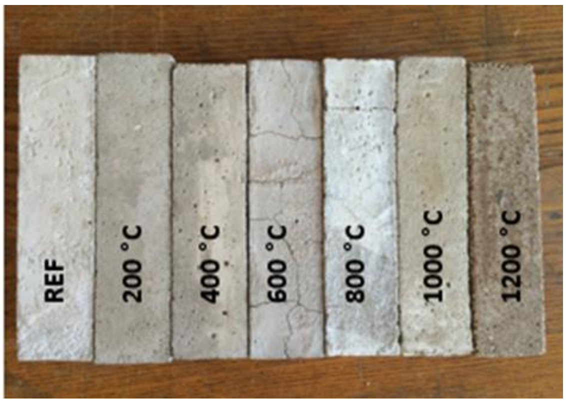

Figure 3. Specimens after thermal degradation.

After initial curing, the specimens were dried at a temperature of 60 °C for two days. Subsequently, the specimens were subjected to gradual heating in a furnace at 200 °C, 400 °C, 600 °C, 800 °C, 1000

°C and 1200 °C (Fig. 3). The temperature increase rate was 5 °C/min. A dwell of 60 minutes at each temperature was provided, in other to find out the effect to temperature on the specimens. After heat treatment, the specimens were left to cool down spontaneously at laboratory conditions.

2.2. Methods

In order to generate the signal, a hammer of 12 g mass, originally suspended from a hanger, was released to fall down on the specimen from a height of 4 cm. The impulse is reflected by the surface but also by micro-cracks and defects of the specimen under investigation. The response was picked up by an MIDI type piezoelectric sensor. Its output voltage was fed into a TiePie engineering Handyscope HS3, which is a two-channel, digital, 16 bits oscilloscope. The piezoelectric sensor was placed at the end of the beam at the center of transverse side and the hammer hit was carried out on the opposite side in the direction of the longitudinal axis. The sensor was attached to the surface of the sample by beeswax. Subsequently, a special smoothing algorithm was used to determine dominant frequencies for each of the output signals. Each measurement run consisted of 5 separate measurements, from which an average was calculated.

Ultrasonic test on concrete is a recognized non-destructive test to assess the homogeneity and integrity of the cement mortar. The natural ultrasound frequency of transducers was 54 kHz. We used the direct method (Fig. 2) which is preferred wherever access to opposite sides of the component is possible. It is essential to make a good acoustical coupling between the concrete surface and the face of the transducer, and this is provided by a medium on water base.

A single harmonic ultrasonic signal method was applied. The measuring apparatus consisted of two principal parts, namely, a transmitting unit and a receiving unit. The transmitting units consists of three functional blocks: a controlled-output-level harmonic signal generator, a low-distortion 100 W power amplifiers and a low-pass output filter designed to suppress higher harmonic components and ensure high purity of the exciting harmonic signal. The main chain of the receiving unit includes an input amplifier with filters designed to minimize the receiving chain distortion and a band-pass filter amplifier. Having been amplified, the sensor output signal is sampled in a DL920 transient recorder, to be subsequently saved in a computer memory for evaluation. As measurement results, we obtained frequency spectra to be subsequently analyzed by means of Fast Fourier Transform.

3. Results and Discussions

A Figure 4 presents the change of dominant frequency of longitudinal waves versus the degree of thermal degradation. Longitudinal waves, which propagate within the sample can affect the mortar element oscillations. The exposure at elevated temperatures causes a decrease of dominant frequency, leading to the conclusion that the material's elastic modulus for each composition also decreases. Predominant frequencies are shifting towards to the lower frequency range in the course of the degradation which is similarly described in [11]. The heating up to 100 °C resulted in the dehydration occurance (conversion of loosely bound to water chemically bound). The Formation Calcium-Silicate-Hydrate (C-S-H) and calcium hydroxide $Ca(OH)_2$ - Portlandite. During further raising temperatures of up to 200 °C begins dehydration cementing compound - the release of bound water for contemporary hydrate decomposition. Between 150 ° C and 170 °C culminates the first stage of decomposition C-S-H and decomposes gypsum $CaSO_4.2H_2O$. These changes did not have a significant influence on the final position of the dominant frequency. At a temperature above 200 °C it led to the release of physically bonded water. Between 250-300 °C, the hydrated cement phases were decomposed and increase of temperature above 300 °C has resulted in decomposition of Portlandite [$Ca(OH)_2 \rightarrow CaO + H_2O$] and the significant formation of micro-cracks. The formation of micro-cracks and Portlandite decomposition result in a 20% reduction of the dominant frequency position. The decrease of dominant frequency is more rapid at the temperature change to 600 °C, where are intense impurities changed. It is seen that the predominant frequencies shifted down towards the lower frequency region, which is caused by phase transition of quartz (in the silicate stone) from triclinic system to the hexagonal system (β at α 573 °C) [12,13]. This results, together with the influence of a difference in thermal expansion disruption bonds between aggregate and cementing compound. In a further step when the temperature was increased to 800 °C were the second phase decomposition C-S-H took place and also decomposition of calcium carbonate [$CaCO_3 \rightarrow CaO + CO_2$], leading to further shifting of the dominant frequency to the lower values [12,13]. The lowest value of the frequency domain, we got through non-carbonation limestone aggregate that produces carbon dioxide (CO_2) - a gaseous substance upsetting concrete as well as around 900 °C, the total decomposition of the cementing compound. For the specimen which underwent a thermal stress by a temperature of 1200 °C is seen that the predominant frequencies shifted upwards towards the higher frequency region. It is evident that a structural change, accompanied by the creation of new crystal phases [Wollastonit β ($CaO.SiO_2$)], takes place in the specimen at temperatures of above 1000 °C.

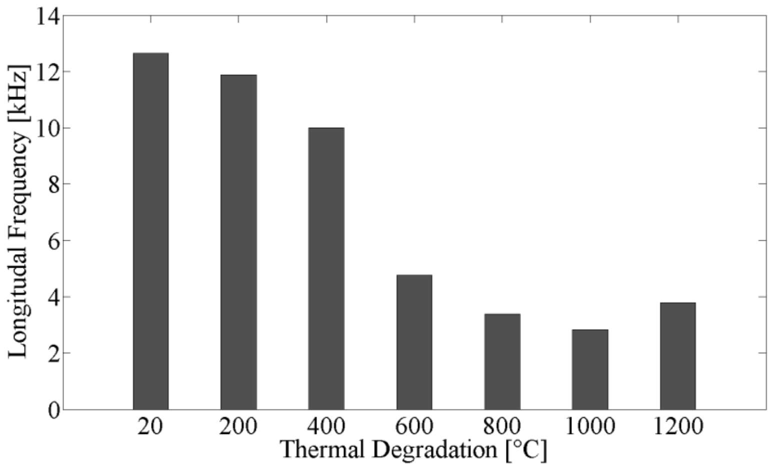

Figure 4. The change of dominant frequency of longitudinal waves versus the degree of thermal degradation.

Figure 5 shows the change of time difference versus the degree of thermal degradation. The time differences were calculated as difference transit time non-degraded specimen and thermally degraded specimen. The pulse transit time between the two transducers is governed by the fastest wave received; which is always the direct wave for a homogenous material. The pulse cannot travel across a material–air interface, but it is able to travel from the transmitter to the receiver by diffraction at the crack edge. Because the travel path is longer than the distance between the transducers, the value of transit time is higher than through sound material. The initial similar values are related with undamaged specimens. But quite steep rise corresponds to the formation of micro-cracks, which do not transmit ultrasonic signals. The last value is connected with the creation of Wollastonite. These results are in very good accordance with impact-echo measurements.

Time of passing of pulses of ultrasound waves was the lowest in the reference samples (REF) s = 38.1 microseconds. During the thermal degradation, increased value of time passage. Upon heating, the samples had values gradual linear tendency to increase up to 400 ° C. At a temperature of 600 ° C has greatly increased transit time up to 98.6 microseconds. It refers to the stage of decomposition of CSH gels. The longest transit time 134.5 microseconds were measured for samples which have undergone thermal degradation at a temperature of 1000 ° C, which corresponds to the beginning of the formation of a ceramic bond. At 1200 ° C, the transit time is reduced to 94.14 microseconds. Which is caused by the formation of β Wollastonite.

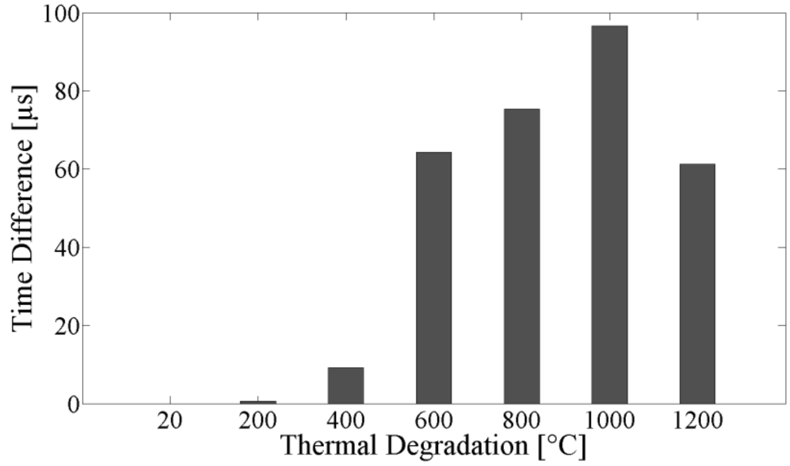

Figure 5. The change of time difference versus the degree of thermal degradation.

Figure 6 shows the capability of the method NUS to detect the occurrence of damage. The results showed that the elastic non-linearity would manifest itself as a change value of amplitude. From empirical evidence, it is clear that micro-cracks in materials are responsible for the enhanced non-linear behaviour. The damping amplitude in specimens is caused microscopic damage as a result of the non-linear constitutive relation at the damage location [14,15]. The non-linear ultrasound spectroscopy has higher sensitivity on smaller damage than impact-echo method. And so, it is visible the relative amplitude (after heating on 600 °C) has the lower values, which is caused by phase transition of quartz (in the silicate stone) from triclinic system to the hexagonal system (β at α 573 °C). In a further step when the temperature was increased to 800 °C were the second phase decomposition C-S-H took place and decomposition of calcium carbonate, which again shows a low relative amplitude value.

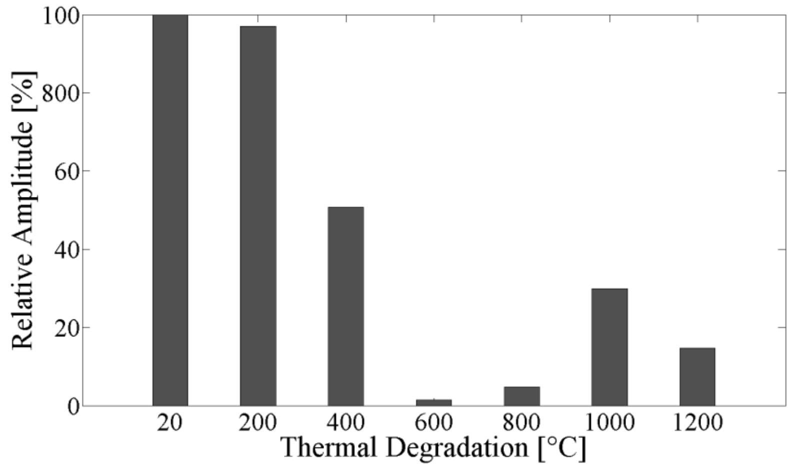

Figure 6. The change of relative amplitude versus the degree of thermal degradation.

4. Conclusions

One key to better understanding the impact of a terrorist attack on a civil construction is to understand the nature of the attack. To that end, different studies about different types of explosions, including physical, chemical, electrical and nuclear were written. These explosions cause various types of damage, some major ones being over pressure, thermal, and projectile. Numerous methods, both analytical and experimental provided the opportunity to understand the impacts from an explosion. Degradation of mechanical properties occurs after concrete is thermally damaged. It is not easy to define the thermal damage in concrete when it is exposed to high temperature.

This paper attempted to compare the results of measurements by the Impact-echo method, ultrasound measurement, and non-linear ultrasound spectroscopy during application on thermal degradation cement mortar specimens. The specimens were intentionally degraded by application of elevated temperatures of 200 °C to 1200 °C. On the basis of the presented measurements and their analysis, the following conclusions can be drawn:

The changes of a dominant frequency towards lower values during increased temperatures excluding the last temperature 1200 °C, probably after the creation of Wollastonite.

The ultrasound, measurement is an evident trend of rising transit time through specimens. We assume that most of these changes can be attributed to crack formation.

The results of Non-linear Ultrasonic Spectroscopy measurements give evidence that the non-linear techniques are extremely sensitive methods to detect small micro-cracks. In our experiments, the non-linearity which was due to the generation of defects caused by the specimen thermal load was reflected in changes of amplitude values.

Furthermore, the Non-linear Ultrasonic Spectroscopy method revealed remarkable sensitivity, showing earlier detection than the Impact-echo method. Therefore, it is concluded that the Non-linear Ultrasonic Spectroscopy method provides a reliable measure to decide on continued use of thermally damaged concrete.

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