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Comparison of results of impedance spectroscopy methods with results of impact-echo method in investigation of high-temperature-degraded concrete

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Abstract

The growth in the transport industry results in more accidents, which often include fire in locations with the concrete structures, which also effects its lifetime and its functionality. After any case of fire loading it is required to verify structure's condition. One way how to assess the risks connected with further usage of the structures after being exposed to the fire is the non-destructive testing. This paper is dealing with non-destructive measurement of changes of electric parameters of the cement based mortars subjected to the high temperatures. Prismatic samples with dimensions of 40×40×160 mm were prepared from water, Portland cement CEM I 42.5 R and quartz sand (in a ratio 1:3), with water/cement ratio 0.46. Samples were intentionally degraded by high temperature in range of 200–1200 °C (200 °C step) and the changes in bulk density, flexural tensile strength and electric properties (loss factor tan δ (f), the imaginary component impedance Im*Z* (*f*) and the real part of component impedance Re*Z* (*f*) were monitored. Two methods were used for testing of electric properties – Impedance spectroscopy and Impact-echo method. The obtained results were compared and suitability of used methods was assessed.

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Keywords: Impact-echo method; cement-based composite material; high-temperature degradation; bulk density; flexural tensile strength; impedance spectroscopy; dielectric losses

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1. Introduction

The infrastructure is expanding very quickly in present time and therefore there is a big demand on building materials. The growth within the automobile industry results in more accidents, which often include fire in locations with the concrete structures, which also effects its lifetime and its functionality.

After any case of fire loading it is required to verify structure's condition. The aim of non-destructive testing is to predict lifetime of a structure and assessment of risks connected with its further usage. Great advantage of this method is that after testing the structure remains undamaged.

The impedance spectroscopy is a non-destructive testing method employing the impedance characteristic frequency dependence to analyse the properties of the cement and non-cement materials. The equipment set-up designed to study the system under investigation includes: a metal-material-metal composition, which is relevant for identifying the application limits of the impedance spectroscopy method. The method cannot be applied to thick-layer low-conductivity materials. Reinforced concrete products may serve as an example. The principle of the mentioned method is based on evaluation of the dielectric studying the dielectric losses versus frequency plots. The dielectric losses of composite materials and plastics can assume values which are many times higher than those of the most materials commonly used in the building industry [4].

Analysis of impedance spectra variances in the tan $\delta(f)$ and Im$Z(f)$ or Re$Z(f)$ of the inhomogeneous materials is a part of the impedance spectroscopy which is still waiting for its development. At present, one is not able to determine unambiguously the individual material component contributions to the total electric conductivity and polarization at various frequencies of the exciting field.

Impact-echo is a method for non-destructive evaluation of concrete structures. The principle of the method is based on analysing an elastic impulse-induced mechanical wave [12]. It is a technique for flaw detection in concrete. The method overcomes many of the barriers associated with flaw detection in concrete based on ultrasonic methods. A short-duration mechanical impact produced by tapping a small steel spherical body gives rise to a low-frequency stress waves that propagate into the structure and are reflected by flaws and external surfaces. A transducer records surface displacements caused by reflections of these waves. Classical impact-echo has receiver attached close to impact. Described method has transmitter in different positions. This signal describes transient local vibrations, which are caused by the mechanical wave multiple reflections inside the structure. The dominant frequencies of these vibrations give information about the condition of the structure or to determine the location of flaws, at which the waves are rebounded. As a rule, the signal is digitized by means of a data processing system to be transferred into a computer memory [12].

2. Material used

Mortar samples (of dimensions 40 mm × 40 mm × 160 mm) were produced using CEM I 42,5 R Portland cement (Ceskomoravský Cement-Heidelberg Cement Group), quartz sand from Filtrační písky, s.r.o. (in a ratio of 1 to 3) and water. (water/cement ratio 0.46). In compliance with CSN 721200 standard, the specimens were demolded after 24 hours, then cured in water for 27 days and finally air-cured for 31 days at laboratory temperature (25±2 °C) and relative humidity of 53±5 %. 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, 1 000 °C and 1 200 °C. The temperature increase rate was 5 °C/min. A dwell of 60 minutes at each temperature was provided, in order to find out the effect of the temperature on the specimens. After heat treatment, the specimens were left to cool down spontaneously at laboratory conditions.

3. Experimental setup

At the Department of Physics, Faculty of Civil Engineering, TU Brno, the IS-based measurements have been implemented using following instrumentation: Agilent 33220A generator, Agilent 54645A double-channel oscilloscope, HP 82350 PCI HP-IB Interface card, and a PC. To operate the above mentioned instruments and to process the IS data acquired, a software called IS_alpha has been prepared by the first author of this paper. [2, 3]

In order to perform impedance analysis, it was necessary to place the samples between brass electrodes. The samples were tested for the frequency spectra from 40 Hz to 1 MHz. Monitored variables were: loss factor tan δ (f), the imaginary component impedance Im*Z* (*f*) and the real part of component impedance Re*Z* (*f*).

In order to generate the acoustic 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, the fast Fourier transform technique was used to transform the recorded waveform into the frequency domain for each of the output signals. Each measurement run consisted of 5 separate measurements, from which an average was calculated. [11,12]

4. Results and discussion

There are 6 curves for different degree of heat stress (0–1000 °C) on the graph of the dielectric loss factor on frequency dependence in Fig. 1.

Curve 8-200A (sample after 200 °C heat stress) has one maximum at frequency 2 kHz and has the highest values of dielectric loss factor on the whole frequency range. The shape of curve is proving of the presence of polarization losses in the material [2].

The sample heated at 400 °C has a smaller maximum at frequencies 70 Hz, then at 800 Hz and shallow peak appears at a frequency of 30 kHz. All graph values dropped significantly compared to the previous sample (200 °C). Polarization losses reduced significantly, which is connected with the water evaporation (dehydration) and degradation of some components of the sealant for the release of water vapor.

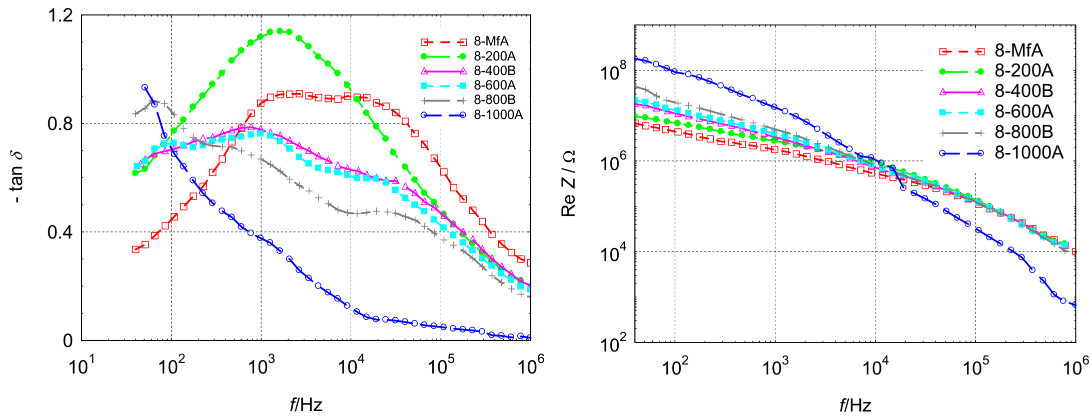

Fig. 1. (a) Spectrum of dissipation factor; (b) Real part of impedance versus frequency of electric field.

A similar pattern shape of the spectrum reaches 8-600A (600 °C) in the lower frequencies but performs a local maximum at frequency of 90 Hz. At the remaining part of the spectrum loss factor values are only slightly lower than of sample 8-400B.

Sample 8-800B annealed at 800 °C shows a pronounced maximum at a frequency of 80 Hz, the value of dissipation factor in the frequency 100 Hz reach higher values than previous samples. Above 100 Hz observed significant decrease of all values of loss factor. Apparently there is a decrease polarization loss, but also to the formation of cracks.

Character of the curve corresponds to sample 8-1000A (1000 °C) suggests the dominance of conduction losses in the material (decreasing character throughout the frequency spectrum). The lack of water and the presence of cracks substantially increased resistance of the sample in the region of the spectrum.

For reference sample 8-MfA (no heat stress), two local maxima in the spectrum of dissipation factor were observed, also at frequency of 2 kHz and 20 kHz, but just to 20 kHz frequency values reach lower dissipation factor than sample 8-200A.

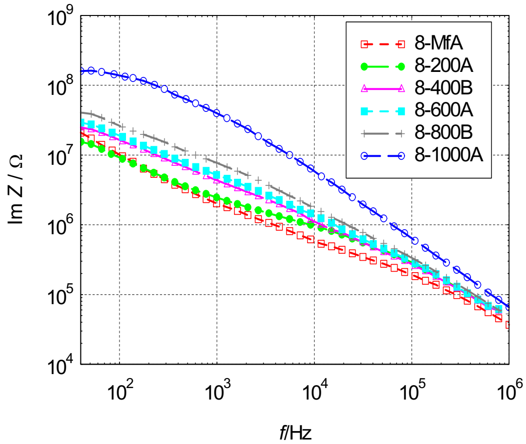

Fig. 2. Imaginary part of impedance versus frequency spectra.

Sample 8-1200A (annealed at 1200 °C) could not be measured; the resistance of the sample is outside the range of measuring instruments (the signal is too attenuated).

The dependency of real part of impedance on the frequency (log-log) spectrum blends for all six samples at a frequency of 10 kHz (Fig. 1). This frequency has the highest value of the real part of impedance for sample 8-1000A and gradually logical descent to the lowest value in sample 8-MFA (no heat stress). A significant portion of the spectrum is in the range of 40-10 kHz. For a frequency of 10 kHz real part impedance values coincides (for samples 0-800 °C), a sample 8-1000A (1000 °C) shows an unexpected drop to as low as 1 kΩ (for 1 MHz).

Graph of the imaginary impedance component dependency on the frequency (log-log) shows that the samples subjected to high temperature stress Im*Z* values are higher in the region of the spectrum (Fig. 2). Curve for the sample without heat stress 8-MFA intersects curve for 8-200A in the 40 - 300 Hz. Subsequently deviates. Reference sample 8-MFA is in the remaining frequency range of the lowest values of the imaginary impedance component.

The Fig. 3a) shoves graph where the increasing value of real and imaginary part of impedance is visible, there are values of all samples for the frequency of 1 kHz. For samples annealed less than temperature of 600 °C, the increase in the values is slow, but rises sharply for the samples annealed above 800 °C. For all samples reached Re(Z) is lower than the values of Im(*Z)*. In the temperature range of 400–600 °C, the shape of the curves is getting changed from concave to convex. The area of this change corresponds to the curve of further investigated dielectric samples at a frequency of 1 kHz, where in the same area has been overturned character of changes. The same shapes and changes has curve of very important peaks of dominant frequencies, for arrangement U1 – S1, for short way of acoustic impulse to sensor for impact echo method. There is small change of values at area I, then big change and then small change in area III.

Permittivity curve (Fig. 3 b) has shape from convex to concave for all spectra. The graph indicated three areas in which we try to capture the structural transformation of materials at given temperatures (stages) of annealing. Separation indicated temperatures up to 400 °C and 573 °C. In this interval are also found inflection points of all curves.

A significant decrease of the dielectric permittivity in area I occurs by dehydration and decomposition of some components of the sealant for the release of steam or CO_2. So most important frequencies of impact-echo (dominant peaks) have very similar shape of decreasing of values. There is one different point of frequency (Imp-Echo method) for the highest temperature of annealing.

In area II we can see no significant changes of permittivity values, at these temperatures, the phase transition of quartz (573 °C) (β-quartz to α-quartz). Decomposition of CSH gel causes compressive deformation of the bond and an increase in porosity.

In the area III dielectric steep drop occurs, which may be related to the emergence of new crystalline phase and a very low strength material.

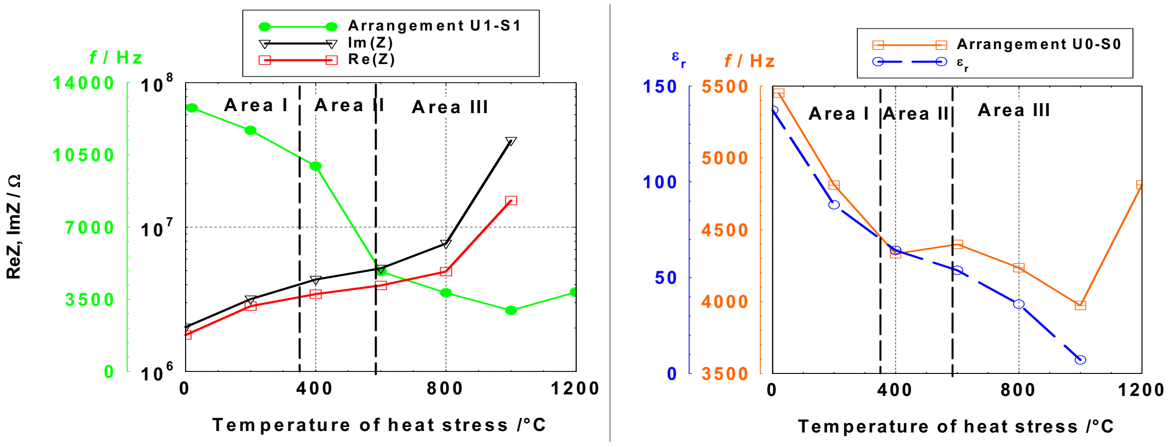

Fig. 3. (a) Discrete values of Im(Z), Re(Z) for 1kHz of electromagnetic field and dominant frequencies for impact echo method, for arrangement U1-S1(short way of wave); for different temperatures of annealing; (b) Values of dielectric permittivity versus frequency of electric field and dominant frequencies for impact echo method, for arrangement U0-S0(long way of wave), for different temperatures of annealing.

During heating, the density of the observed samples decreases. Heating of the samples leads to dehydration and decomposition of some components of the sealant for the release of steam or CO_2. The largest decrease in density occurs at the beginning of heating (to the temperature of 400 °C).

Also the tensile strength of the samples decreases with increasing temperature, which is related to dehydration and decomposition of CSH gel, but also with the phase transformation of quartz at 573 °C (β-quartz to α-quartz). Decomposition of CSH gel causes the pressure connected with deterioration of the bonds with subsequent increase of porosity. At temperature above 1000 °C, crystalline phase is formed in the specimens.

5. Conclusion

The obtained results show that the frequency inspection carried out by means of the Impact-echo method makes a convenient tool to assess the quality and life of these composite materials when exposed to elevated temperature. The acoustic results confirmed the differences in the structure of mortar specimens. The tested specimens were mortars prepared from a mixture of cement and quartz sand. The specimens were intentionally degraded by application of elevated temperatures of 200 °C to 1 200 °C. A shift of the predominant frequencies and a change in the damping coefficient were observed to occur during the degradation process.

Specimens were tested by impedance measurement and interesting similarity were obtained with impact echo test results. When dominant frequencies were decreasing, the permittivity was decreasing too, and by the same trend. The deviation was observed for the highest temperature of annealing. Difference between measured results of arrangement U1-S1 (short way) and U0-S0 (long way of wave) are in width of interval of frequencies and in numerical rank. Next discussion is necessary for understanding of prove of degradation of annealing concrete.

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References

[1] I. Kusak, M. Lunak, Comparison of impedance spectra of concrete recorded with utilizing carbon transition paste, Adv. Mat. Res. 897 (2014) 131–134.

[2] V. Mentlik, Dielektrické prvky a systémy, BEN – technická literatura, Praha, 2006.

[3] M. Lunak, I. Kusak, Z. Chobola, Dielectric properties of concrete specimens after heat stress, Appl. Mech. Mater. 446–447 (2014) 1389–1394.

[4] M. Cabeza, P. Merino, A. Miranda, X.R. Novoa, I. Sanchez, Impedance spectroscopy study of hardened Portland cement paste, Cement Concrete Res. 32 (2002) 881–891.

[5] T. Ficker, L. Topolar, I. Kusak, Is componential strength analysis of concrete possible?, Mag. Concrete Res. 65 (2013) 1480–1485.

[6] L. Pazdera, L. Topolar, V. Bilek, J. Smutny, I. Kusak, M. Lunak, Measuring of Concrete Properties during Hardening, ESA 2010. 1. CZ, Palacky University (2010) 311–318.

[7] I. Kusak, M. Lunak, P. Schauer, Tracing of concrete hydration by means of impedance spectroscopy (New tool for building elements testing), Appl. Mech. Mater. 248 (2013) 370–378.

[8] J.R. Macdonald, Impedance spectroscopy, emphasizing solid materials and systems, bibliography, Canada, 1987.

[9] M. Lunak, I. Kusak, L. Pazdera, L. Topolar, V. Bilek, Monitoring of cement-based material solidification, focusing on electrical properties, Proc. of the 48th International Scientific Conference Experimental Stress Analysis 2010, 233–240.

[10] M. Krause, M. Barmann, R. Frielinghaus and et al., Comparison of pulse-echo methods for testing concrete, NDT & E International, 30 (1997) 195–204.

[11] P. Mazal, L. Pazdera, J. Dvořáček, Application of acoustic emission method in contact damage identification, Int. J. Mater. Prod. Tech. 41 (2011) 140–152.

[12] I. Plskova, Z. Chobola, M. Matysik, Assessment of ceramic tile frost resistance by means of the frequency inspection method, Ceram.-Silikaty 55 (2011) 176–182.

[13] J.M. Torrents, J. Roncero, R. Gettu, Utilization of impedance spectroscopy for studying the retarding effect of a superplasticizer on the setting of cement, Cement Concrete Res. 28 (1998) 1325–1333.