The assessment of mechanical properties of thermally stressed composite materials using radio frequency electrical field

I Kusak¹, M Lunak¹, R Dvorak¹ and Z Chobola¹

¹ Institute of Physics, Faculty of Civil Engineering, Brno University of Technology, Veveří 95, 602 00 Brno, Czech Republic
Email: kusak.i@fce.vutbr.cz

Abstract. The main topic of this paper is the testing of cement-based composite materials that were stressed by high temperature. Impedance spectroscopy has been used as a non-destructive measurement method. The specimens were made from mortar mixture of CEM I type Portland cement, silica sand and fine gravel of various size classes for two sets of specimens. The sets, A and B, differed from each other also by the plasticizer used. The specimens were exposed to high temperatures in the range 23–1200°C. Studying the structural changes in the process of heat build-up is an efficient way to assess the reliability of whole construction. Using the impedance spectra the relative permittivity, electrical conductivity and other characteristics have been obtained. In accordance with Debye’s theory of dielectrics the changes of impedance parameters have been put into context with structural changes. For higher range of frequencies, 100 MHz up to 3 GHz, R&S ZNC vector analyzer with DAK 12 coaxial probe manufactured by Speag has been used. In this frequency spectrum the electrical conductivity and relative permittivity as a function of frequency have been measured.

1. Introduction

If concrete is exposed to elevated temperatures, its mechanical and chemical properties, such as the allowable tensile and compressive stresses, the modulus of elasticity, the Poisson’s ratio and some other quantities, will undergo a change. These changes depend on the temperature change rate, temperature extreme values, fire duration and concrete type [1, 2].

Thanks to its excellent resistance to compressive forces and to its high durability, concrete is widely used as the main structural material in advanced structures. However, concrete exhibits some shortcomings. One of them consists in its low toughness, or, in other words, its brittleness, due to which concrete structures may collapse suddenly or even in an explosive way. The seriousness of this problem arises with the concrete strength and the structure size. If the ultimate strength is exceeded, even locally only, a disaster may occur without previous warning in the form of a plastic deformation preceding the breakdown. High temperatures enhance the magnitude of this problem. There is a number of large concrete structures, such as TV towers, dams, bridges, nuclear power plant containments, which may be exposed to elevated temperatures, for example, due to fire, a nuclear disaster, or an act of terrorism. The elevated temperature exposure may be either shock-type (quick heating or cooling) or long-time. Concrete structures may be exposed to elevated temperatures in consequence of fire, explosions or chemical reactions. When concrete is heated to a certain temperature, C-S-H gel dehydration, thermal incompatibility between aggregates and cement grout, and porous pressure in the cement grout will take place, being the main harmful factors [3, 4, 5].
Thermal stress induced structural change monitoring is of high importance for the determination of the overall structural reliability. The present paper focuses on the study and applicability of the impedance spectroscopy to the testing of cement-based composites of cement mortar, aggregates and quartz sand mixes, which have been realized in the framework of this research and were degraded while being exposed to elevated temperatures (200°C – 1200°C).

2. Experiment setup
For our research we have prepared concrete specimens with dimensions 0.1 m × 0.1 m × 0.4 m.
Designed mixtures are at Table 1. Consistency 550 mm flow a cone. The specimens were soaked in water for 28 days and then dried at first at the laboratory temperature and then for 48 hours in a ceramic furnace at a temperature of 110°C. The concrete specimens were heated in Rhode KE 130B programmable laboratory furnace at the heating rate of 5°C/min. Selected temperatures T = 200°C, 400°C, 600°C, 800°C, 1000°C and 1200°C were maintained for 60 minutes.
The principal difference in the composition of B-type specimen consists in the use of a lower grading, namely, 11/22, and, at the same time, in the plasticizer used.
For higher range of frequencies, 100 MHz up to 3 GHz, the vector analyzer R&S ZNC has been used with the connected coaxial probe DAK 12 manufactured by Speag. In this frequency spectrum the electrical conductivity and relative permittivity as a function of frequency has been measured.

| Compounds                        | Amount of each compound for 1 m³ [kg] |
|----------------------------------|--------------------------------------|
|                                  | Mixture A    | Mixture B    |
| Cement CEM I 42.5 R              | 345          | 345          |
| Fine aggregate Žabčice 0/4 mm    | 848          | 896          |
| Coarse aggregate Olbramovice 8/16 mm | 980          | 521          |
| Coarse aggregate Olbramovice 11/22 mm | -            | 391          |
| Superplastizer Sica Viscocrete 2030 | 2.8          | 2.5          |
| Mix water                        | 160          | 173          |

The compressive strength was measured in compliance with EN 12390-3:2009 Testing hardened concrete - Part 3: Compressive strength of test specimens. The compressive strength test was carried out on broken ends of the beam, on which a flexural tensile strength test had been previously performed. Steel fixtures for the upper and lower pressure application areas were used to ensure an accurately defined area of 100 × 100 mm. The compressive force was applied perpendicularly to the compaction direction.
The tensile strength was measured in compliance with EN 12390-5:2009 Testing hardened concrete testing - Part 5: Flexural tensile strength of test specimens. The tensile strength test was carried out on beams by means of a four-point bending. The bending force was applied perpendicularly to the compaction direction of the test specimens [6, 7].

3. Results
The electric conductivity of A and B-formula specimens was measured by means of a vector analyser in the frequency range from 100 MHz to 3 GHz. The readings ranged from 10⁻⁶ S/m to 0.15 S/m. Growth trends were established, however, they were not uniformly distributed over all of the formulas and frequencies. Slopes of the lines were calculated, however, they were not uniformly distributed over all of the formulas and frequencies.
For the A-formula specimen set Figure 1, a steep growth takes place up to the frequency of 500 MHz. Subsequently, a linear rise follows up to 2350 MHz, where a steeper increase sets on again. The readings are oscillating around the level, which shows the linear trend with the mentioned nickpoint.

The highest electrical conductivity values were found on the specimen which has not been loaded thermally. Lower values correspond to the specimen which was dried up at 200°C. Its conductivity gets close to that of the 20°C specimen at the highest frequency. A noticeable decrease in value occurred at 400, 600, 800°C, where the differences in conductivity values narrow. A further drop was shown – as expected – by the specimens, which had been heated to 1000°C and 1200°C. Their conductivities differed from each other at 2356 MHz only. The above mentioned frequency value is approximate only and cannot be specified accurately. It is rather a region.

![Figure 1](image1.png)

Figure 1. Electrical conductivity of B-formula concrete specimens, as measured by ZNC vector analyser.

![Figure 2](image2.png)

Figure 2. Electrical conductivity of B-formula concrete specimens, as measured by ZNC vector analyzer.
The conductivity plots of the B formula specimens Figure 2 are similar in shape. We also observe the rapid change of slope, however at a lower frequency than above – at about 2 450 MHz. The highest conductivity values are reached by no-thermal-stress subjected specimens. However, the conductivities of other specimens are below those of the A-formula specimens. The B-formula specimens contain higher grading aggregates (11/22 Olbramovice), the same amount of cement but a lower amount of water. Due to the thermal stress, water is released on a bigger scale, because of the lower binder amount and, consequently, lower number of conducting paths from the C-S-H gel. The latter disintegrates in a larger extent, too. At higher temperatures, the conductivity change is caused by the concrete structure transformation to the vitreous phase.

Figure 3. Compressive and flexural tensile strengths for A-formula specimens under stress. Figure 4. Compressive and flexural tensile strengths for B-formula specimens under stress.

The B-type specimens exhibit higher compressive strength $f_c$ and flexural tensile strength $f_{ct}$ values for the mentioned thermal stress variants. The B-type specimens contain higher grading aggregates. Lower conductivities than at the A-formula are only observed at 1 000°C and 1 200°C.

Lower permittivity values Figure 5 of the A-formula specimens are observed in the dielectric constant spectra at frequencies ranging from 100 MHz to 3 GHz. These spectra show a decreasing trend up to 1 GHz to oscillate around a single value. They show a decline in the vicinity of 3 GHz. The higher degree of firing results in a lower value of the permittivity, which is the same as with the B-formula specimens, where, however, the values corresponding to 400°C and 600°C have been unexpectedly interchanged. At 1 000°C and 1 200°C, the permittivity values have been interchanged, too. Only these, last-mentioned changes and their trend are in accordance with the increase of $f_c$, $f_{ct}$ for 1 200°C as against 1 000°C.

Figure 5. Relative permittivity of A-formula specimens under stress.
When comparing the spectra of Figures 5 and 6, we observe a larger scattering (apparently waviness) of the measured points for B-formula specimens, which contain coarser aggregates. The waviness of the spectrum shape for the B-formula specimens is due to the behaviour of the granite grade, which is present in the higher grading broken stone from Olbramovice. The granite grade used has a higher content of polarizable particles, whereas the cementing compound contributes to the conductivity.

4. Conclusion
In the present paper, two sets of concrete beams, which had been subjected to thermal stresses of various intensities, were successfully tested by means of a vector analyzer in the frequency range from 100 MHz to 3 GHz. This unique measurement method is non-destructive. In spite of that, the DAK contact probe measurements provided us with conductivity and permittivity spectra.

The conductivity and permittivity values decreased when the annealing degree increased, which is in agreement with the concrete structure metamorphosis into a vitreous phase. For comparison, spectra showing the decreasing concrete strength versus the growing annealing degree were presented. Downward trends were compared with a slight increase resulting from the specimens' transition to the vitreous phase.

In this experiment, the analyzer, being equipped with a Speag probe, proved to be a fast-operating, non-destructive tool, which has been underutilized in the NDT practice thus far. Monitoring of impedance parameters being in correlation with material changes is very important for evaluation of structural reliability.

Acknowledgments
This paper has been worked out under the project GAČR No.16-02261S supported by Czech Science Foundation and project No. LO1408 "AdMaS UP - Advanced Materials, Structures and Technologies", supported by Ministry of Education, Youth and Sports under the „National Sustainability Programme I“. 

References
[1] Monica J H and Harris A T 2013 Progress in Materials Science 58 pp 1056–1102
[2] Saafi M, Andrew K, Mcghon D, Taylor S, Rahman M, Yang S, Zhou X and Tang P L 2013 Construction and Building Materials 49 pp 46–55
[3] Lhoták P 2014 http://www.uochb.cas.cz/Zpravy/PostGrad2004/7_Lhotak.pdf
[4] Matsutani S, Shimosako Y and Wang Y 2012 Physica A 391 5802
[5] Fernandez-Jimenez A 1999 Cement Concrete Res. 29 1313
[6] Kusak I, Lunak M and Schauer P 2013 *Appl. Mech. Mater.* **248** 370

[7] Tupy M, Sotiriadis K, Kusak I, Lunak M, Stefkova D and Petranek V 2015 *J. of Mater in Civil. Eng.* **28** 04015171