IMPEDANCE SPECTROSCOPY – NEW TOOL FOR TESTING CERAMIC ROOFING TILES et al. BUILDING MATERIALS

IMPEDANČNÍ SPEKTROSKOPIE JAKO NÁSTROJ PRO SLEDOVÁNÍ KERAMICKÉ TAŠKY A JINÝCH STAVEBNÍCH MATERIÁLŮ

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Abstract
A NDT impedance spectroscopy method has been used to characterize ceramic roofing tile specimens. Differences between dry and various water-content condition specimen tanδ(f) spectra variances have been observed. Furthermore, based on the predominant loss type, the material quality has been characterized. The method reproducibility has been verified by multiple measurements.

Keywords: *impedance spectroscopy, dielectric losses, polarization losses, conductivity losses, roofing tile*

Abstrakt
NDT metodou impedanční spektroskopie byly charakterizovány vzorky keramických tašek. Byly pozorovány rozdíly ve spektru tanδ(f) vzorků v suchém stavu a různých stavech absolutní vlhkosti. Dále byla popsána kvalita pomocí druhu ztrát dominujících v materiále. Měřením byla ověřena reprodukovatelnost této metody.

Klíčová slova: *impedanční spektroskopie, dielektrické ztráty, polarizační ztráty, vodivostní ztráty, keramická taška*

1. Introduction

The method presented here is based on the measurement of electric impedance characteristics of the system whose configuration is **metal - building material - metal**. The building material behaves as a **dielectric** in this system. We are therefore studying the properties of this dielectric. The key indicators, representing the specimen system to be analyzed are the measured system impedance real and imaginary part and, mainly, the **loss factor** versus frequency plots. As supplementary indicators serve the resistance and capacitance versus frequency plots of the system into which the building material in question was built in.

The method is intended to provide both qualitative and quantitative evaluation of the above-mentioned parameters. The curve shape makes it possible to detect the effect of **dampening and drying** on the material, **changes in its structure, change in porosity**, its **service life degradation**, **technology related defects**, etc. By comparing the results with an impedance spectrum of defined specimens, we may exactly identify the deviations in the properties being tracked. However, it is beyond the capacity of this method to quantify the building material parameters. On the other hand, this method can be very well used as a comparative method and we expect that, after some analysis and refinement of the idea have been accomplished, it can be used as an absolute method.

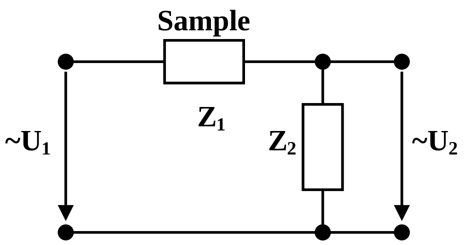

Fig. 1. Circuit diagram for the unknown impedance Z_1 to be measured and the known value impedance Z_2.
Here U2, U1 = input, output electric voltages, respectively.

Water constitutes a substantial part of the structure. Depending on the ways water is bonded in the material, several types may be distinguished:

Chemically bonded water: It can only be released by the action of higher temperatures (200 to 700 °C).
Physically bonded (capillary) water: It is absorbed on fine particles' surface. Its amount depends on the content of the finest particles and on the environment moisture.
Free water: It is contained in cavities and pores. It is the easiest to evaporate. Its content decreases in time. The pores it is leaving behind reduce the material compactness and strength.

2. Roofing tile specimen parameters

Material:	fired
Surface treatment:	unplastered
Colour:	red hues
Mass per m^2:	64 kg
Type:	plain tile
Width:	180 mm
Length:	380 mm
Volume:	cca $8.208 * 10^{-4}$ m^3
Mass	cca 1.5 kg
Safe pitch angle in degrees:	30
Consumption per m^2	36 pcs.

Table. 1. Roofing tile specimen parameters

3. Roofing tile specimen description

The tile ranks among the oldest and most important roofing elements. It is a smooth-surface roofing element, which can be laid down on so-called dense or open lathing (i.e., imbricate or crown-shaped overlapping). It can be used to cover various, even oval-shaped, building elements (bull's eyes, cones, roof valleys).

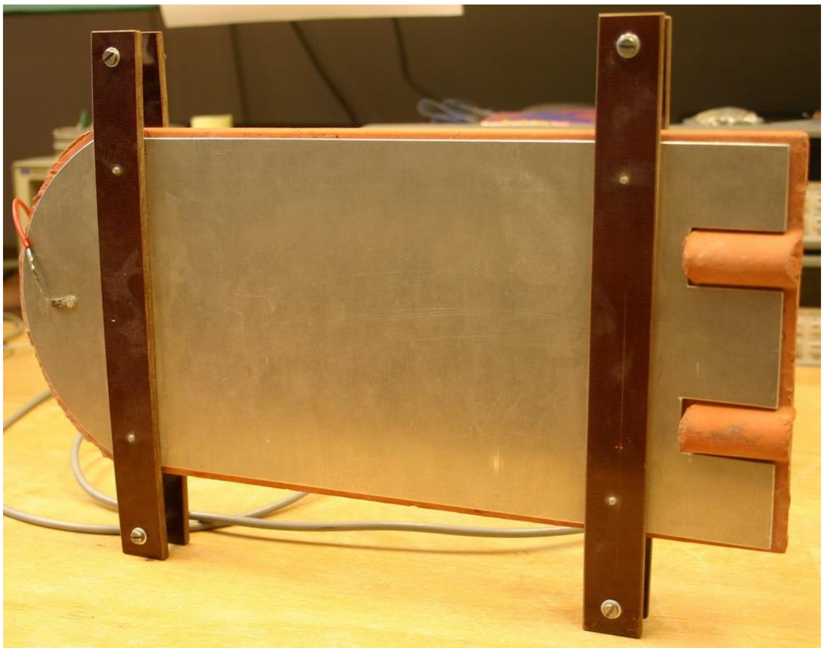

Fig. 2. Roof tile

4. Experiment and humidity cycles

The objective of this experiment consisted in distinguishing between the ceramic tile impedance spectra prior to and in the course of the humidity cycle. The specimens under investigation were inserted between two electrodes (which were pressed against the specimens using a screw fixture – see the photo) and subsequently subjected to the impedance analysis.

5. Measurement results

In the diagram (see Fig. 3), the loss factor (tan delta) versus frequency (*f*) plot is shown, the frequency as well as the loss factor being plotted in a logarithmic scale for lucidity.

In the dry tile spectrum, the upward-sloping loss factor curve suggests that the polarization losses are predominant up to a frequency of 1,1 kHz. What follows above this frequency (downward-sloping loss factor curve) corresponds to the conductivity losses being predominant in the tile (ceramics). From the model premises it follows that good-quality materials shall show no or minimum polarization losses as these losses are signalling a water content, inhomogeneities, graininess, loss of elasticity or structure deterioration being present in the material.

Starting from the frequency of about 10^5 Hz, the loss factor curves for dry tiles (prior to soaking) as well as those for a tile after a five-day drying interval show a decreasing trend (the values are fading into one another). Unfortunately, no conclusions concerning the kind of the losses in the material can be drawn as the loss factor values are below the method accuracy.

The curves are featuring 2 loss factor peaks (local maxima) (in the region from 40 to 10^4 Hz) for various absolute humidity (water-content) conditions. It can be assumed that the post-one-drying-day spectra will show two peaks in the low-frequency region, too.

At the same time, the peak frequency is observed to shift, i.e., it is going down with increasing water content. This tendency is contrary to that we had observed in “cetris” wood-cement chipboards at various absolute water-content conditions [6].

	Specimen mass **m/kg**	Absolute humidity **Φ / kg.m^{-3}**	Specimen electric resistance at 1000 Hz **R/ MΩ**	Specimen capacitance at 1000 Hz **C/pF**
Dry specimen	1,4675	-	2,414	203,8
1 drying day	1,5120	**54,216**	0,161	471,6
2 drying days	1,5025	**42,641**	0,222	494,5
5 drying days	1,4680	**0,609**	2,267	218,3

	Absolute value of impedance **|Z|/kΩ**	Phase angle **-φ/°**	Electrical conductivity at 1000 Hz **G/ μS**	Relative permittivity at 1000 Hz **ε_r**
Dry specimen	743	72,30	0,4149	5,2
1 drying day	144	25,45	6,2460	12,1
2 drying days	183	34,82	4,4670	12,7
5 drying days	695	72,25	0,4416	5,6

Table. 2. Electrical properties of a roofing tile specimen in the humidity cycle

Absolute water content (absolute humidity) in the specimen $\Phi = m/V$, where m is the mass increment, V, the volume.
The slight difference (rather an increase) which was observed after the first and second drying day can be attributed to the effect of water molecules on the surface of the ceramics specimen. This influenced the electric quantity measurements using an RLCG bridge, which is, in principle, an AC measuring instrument. Subsequent measurements are already providing a true picture of the roofing tile bulk properties. The precondition that the specimen growing capacitance together with decreasing resistance should result in reducing the impedance absolute value therefore appears to be met here.

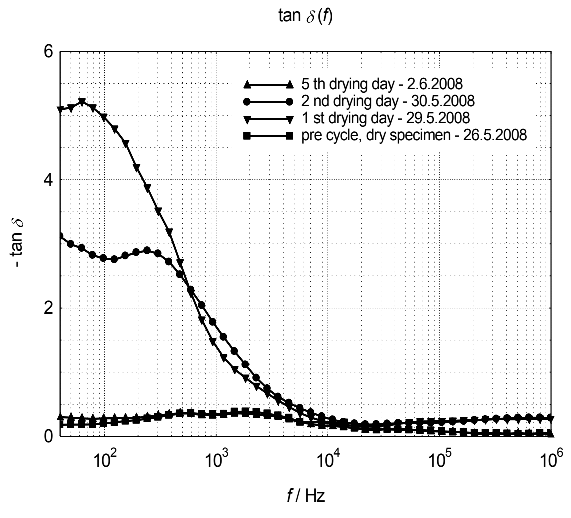

Fig. 2. Roofing tile loss factor versus frequency for various water contents

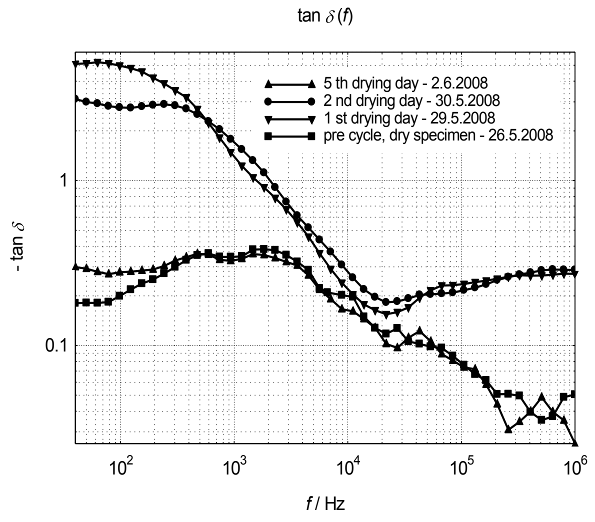

Fig. 3. Roofing tile loss factor versus frequency for various water contents (both frequency and loss factor are plotter in log scale)

The specimens under test, while dry, were also subjected to reproducibility tests several times at various time periods. Thus obtained curves proved to almost duplicate each other with minimum variances, thus giving evidence of a good reproducibility of the method.

5. Conclusion

The impedance spectroscopy method has been used to characterize the fired plain roofing tile specimens.

The resulting change in the frequency characteristics fits the assumption that the respective physical property changes are reflected in the impedance loss factor. Dielectric losses have been described. The method reproducibility has proved to be very good.

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