Noise Spectroscopy Tests in the Analysis of Materials and Periodic Material Structures

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Abstract— The authors discuss the application of a broadband noise signal in the research of periodic structures and present the basic testing related to the described problem. Generally, noise spectroscopy tests are carried out to verify the behaviour of the response of periodic structures, and the related objective consists in recording the properties of microscopic structures in natural and artificial materials. The aim is to find a metrological method to investigate structures and materials in the frequency range between 100 MHz and 10 GHz; this paper therefore characterizes the design of a suitable measuring technique based on noise spectroscopy and introduces the first tests conducted on a periodic structure. In this context, the applied instrumentation is also shown to complement the underlying theoretical analysis.

1. INTRODUCTION

In general terms, spectrometry can be defined as a discipline analyzing the properties and origination of the spectra of harmonic signals or electromagnetic waves. The related research methods are based on the interaction between an electromagnetic wave and the measured sample of matter. The dependence of irradiation intensity changes on the wavelength is examined within electromagnetic spectroscopy; this discipline comprises, for example, Raman spectroscopy, which measures the spectrum of electromagnetic irradiation scattered on the basis of the Raman effect (non-elastic scattering). This effect causes the scattered irradiation to exhibit a wavelength slightly different from the incident radiation, mainly due to a part of the energy being transferred at vibrational junctions of a molecule. The discussed technique provides information on the structure and spatial arrangement of the quantum mechanical model of a molecule, and it is applicable for different materials, such as carbon [2, 3]. Another subregion of spectrometry consists in a method which utilizes the Fourier transform and is based on a mathematical transformation of the interferogram of the signal intensity dependence on the path difference of the beams; this difference is acquired via detecting the signal out of the interferometer. The interfering beams travel through the burette containing the sample. Fourier type spectrometers [1] are applied in, for example, infrared spectroscopy, UV/VIS spectroscopy, attenuated total reflection, atomic absorption spectroscopy, X-ray fluorescence, and weight spectroscopy to measure the proportion of weight to the ion charge. Weight spectrographs and spectrometers utilize the motion of a particle of the quantum mechanical model of matter in the electric and magnetic fields. We have the formula

$$m = \frac{p^2}{2 \cdot E_{\text{kin}}}, \tag{1}$$

where m is the weight, p are the dynamics of a moving particle, and E_{kin} is the kinetic energy of a specific particle. The weight can be determined from a comparison of the dynamics and the kinetic energy; this is performed via the passage of an ion having the charge Q through the "energy filter" and the "dynamics filter", both of which are materialized by means of an electric and a magnetic field from the application of Faraday's law of induction and Lorentz's forces acting on the particles of mass:

$$F = q\,(E + v \times B)\,, \tag{2}$$

where $\mathbf{F}$ is the vector of the force acting on the particle with an electric charge q, $\mathbf{E}$ is the electric intensity vector, $\mathbf{v}$ is the vector of the instantaneous velocity of the motion of the electrically charged particle, and $\mathbf{B}$ is the magnetic flux density vector. Other subareas comprise spectroscopy utilizing nuclear magnetic resonance, which is applied to determine the distribution of atoms in the vicinity of nuclei exhibiting non-zero nuclear spin (^{1}H, ^{13}C, ^{31}P, ...). In the given context, let us

note that nuclear magnetic resonance spectroscopy [4–6] is a physical-chemical method exploiting the interaction between atomic nuclei of the quantum mechanical model of matter and an external magnetic field. The technique examines the distribution of nuclear spin energies in the magnetic field and investigates the transition between individual spin states caused by radio frequency irradiation. Current methods within NMR spectroscopy are nevertheless fully applicable also in defining the spatial structure of smaller proteins, thus complementing the X-ray structural analysis. Another related technique utilizes electron paramagnetic resonance to measure particles containing non-pair electrons; this is a method enabling us to solve the problems of magnetic resonance spectroscopy. Other procedures involve nuclear quadrupole resonance and muon resonance [7]. Noise spectroscopy can be effectively practised via both harmonic analysis and statistics. To evaluate signals in continuous time, we can suitably use the Fourier transform [8], which can be further modified for other signal types. The evaluation of discrete signals is then feasible by means of the discrete Fourier transform [9] and the fast Fourier transform algorithm [10]. However, the Fourier transform is not applicable for the investigation of non-stationary signals; such signals can be examined more advantageously via wavelet transform and its modified algorithm, the discrete wavelet transform [11, 12]. Noise spectroscopy also utilizes wideband signals, for which we can suitably use the Burg algorithm method [13–15].

1.1. Ultrawideband Signals (Noise)

Noise can be generally characterized as a stochastic, random signal, whose description is deliverable via several approaches. Of these, the most prominent one consists in the amplitude area, where the signal is described by distributing the amplitudes, expressing the probability density, and specifying the distribution function. Another description technique is materialized via the autocorrelation function in the time region and by means of the power spectral density in the frequency area. Noise can be classified with "colours" assigned according to the frequency range; we then distinguish between white, grey, brown, red, pink, and other noise variants. These noise types are specific in their properties and sources. White noise, for example, consists of random samples exhibiting uniform and constant spectral power densities. Other properties of white noise include also infinity of the frequency spectrum; this is, however, a mere theoretical assumption because if the frequency spectrum were infinite, the total power of the signal would be infinite too. By extension, pink noise — also known as *1/f noise* or *oscillating noise* — is a signal or process having such frequency range that the power frequency density is directly proportional to the reverse value of the frequency. This condition occurs multiple times within the process and is therefore termed *transition between white and red noises*. Red noise is similar to the pink one but exhibits a power frequency density lowered by 6 dB per octave with increasing frequency [18].

1.2. Macroscopic Environment

The term *macroscopic material* generally denotes any material observable with the naked eye. The denomination is related to longitudinal dimensions, although it is also used to denote the effects and quantities connected with macroscopic objects. An isotropic environment constitutes a form of matter whose physical properties are identical or approximately identical in all directions of the coordinate system; an anisotropic environment is then one whose properties change in directions of the coordinate system. Then, for example, the relationship between the electric flux density $\mathbf{D}(t)$ and the electric intensity $\mathbf{E}(t)$ can be expressed by linear combination of the squared component of the vector $\mathbf{D}(t)$:

$$\begin{pmatrix} D_x \\ D_y \\ D_z \end{pmatrix} = \begin{pmatrix} \varepsilon_{11} & \varepsilon_{12} & \varepsilon_{13} \\ \varepsilon_{21} & \varepsilon_{22} & \varepsilon_{23} \\ \varepsilon_{31} & \varepsilon_{32} & \varepsilon_{33} \end{pmatrix} \begin{pmatrix} E_x & E_y & E_z \end{pmatrix} . \begin{pmatrix} B_x \\ B_y \\ B_z \end{pmatrix}$$
$$= \begin{pmatrix} \mu_{11} & \mu_{12} & \mu_{13} \\ \mu_{21} & \mu_{22} & \mu_{23} \\ \mu_{31} & \mu_{32} & \mu_{33} \end{pmatrix} \begin{pmatrix} H_x & H_y & H_z \end{pmatrix} \begin{pmatrix} J_x \\ J_y \\ J_z \end{pmatrix} = \begin{pmatrix} \gamma_{11} & \gamma_{12} & \gamma_{13} \\ \gamma_{21} & \gamma_{22} & \gamma_{23} \\ \gamma_{31} & \gamma_{32} & \gamma_{33} \end{pmatrix} \begin{pmatrix} E_x & E_y & E_z \end{pmatrix} . \quad (3)$$

The coefficients ε_{jk} of this linear transformation (3) are components of the symmetrical tensor of electric permittivities. A homogeneous environment is defined as one having constant properties in the space of the monitored material. Similarly, (3), it is possible to write the magnetic field properties, namely the magnetic permeability μ_{jk} with the magnetic field intensity vectors $H(t)$, the magnetic flux density $\mathbf{B}(t)$, and the current field with the specific conductance γ_{jk} (3) for vectors of the electric field intensity $\mathbf{E}(t)$ and the current density $\mathbf{J}(t)$.

1.3. Periodic Structures

From the perspective of description, a macroscopic material (MM) can be defined via a quantum mechanical (QM) model. The MM is then examined based on the incidence/irradiation of an electromagnetic wave, and from this interaction we then deduce the properties of the sample. The QM model of the sample comprises a high number of repeated structures, and it is thus possible to use the term *periodic material structure*. Depending on the result of the EMG wave interaction, we can deduce the basic and complementary properties of the sample (conductor; semiconductor; or insulator). Such utilization of similar effects is also typical of spectroscopy [2]. Research in the given area was already performed by Yablonovitch, whose experiments focused on an EMG wave in the spectrum of light [19]. The beginnings of research into the interaction between radiation and a periodic structure can be traced to the first years of the 20th century, a period when Bragg discovered by observation that, under certain conditions, atomic structure can behave like a mirror. This holds true, for example, in X-rays if the conditions are satisfied of a wavelength λ and a distance d between two neighbouring atoms at the angle of incidence Θ:

$$\lambda = 2d \cdot \sin(\Theta \pm \delta), \tag{4}$$

where δ is the angle deviation. Reflections of the incident EMG wave will occur, Fig. 1.

It is nevertheless obvious that the material and its atoms in themselves do not exhibit this property and that the periodicity of the structure must be ensured on the scale of wavelengths of the incident EMG wave. In periodic structures, the above-described effect can be used to determine the properties of the monitored sample of material. If an unknown sample of material, conceived according to the QM model, is irradiated with an EMG wave exhibiting a sufficient wavelength, we will find conditions that facilitate reflection of the selected EMG wave from the applied electromagnetic wave spectrum. The wave selection depends on the actual periodicity of the material. The ideal frequency range of the transmitted wave is infinite bandwidth; theoretically, this condition can be satisfied by white noise. When the reflected part of the EMG wave is captured, it is advantageous to have ready suitable evaluation tools, for example the Fourier transform, wavelet transform, or other techniques introduced above.

1.4. Periodic Structure Analysis

In order to be able to investigate the actual spectroscopic method working with a white noise signal, there has to be the possibility of mathematically describing periodic structures, and it is important to know their mathematical characteristics. The structure can then be analyzed. If we assume a periodic structure with finite dimensions, such analysis is performable with a finite method enabling basis approximation of the geometry and the function. In periodic structures exhibiting a low level of periodicity and large dimensions of the model, finite methods cannot be applied effectively, and a different mathematical model must be chosen. Artificial periodic structures include, for example, metamaterials [14] composed of elementary bound resonators.

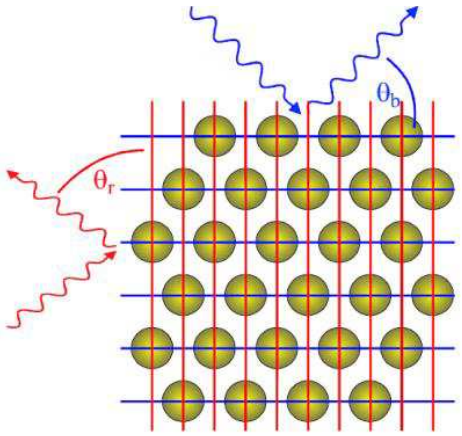


Figure 1: X-ray reflection from the periodic structure of atoms.

Figure 2: The applied noise generator and power amplifier. The image shows the tested noise generator, whose output power is 0 dBm in the frequency range of between 100 kHz and 10 GHz.

1.5. Benefit of Noise Spectroscopy

The contribution of noise spectroscopy consists in the use of an ultrawideband signal, which will enable us to acquire, within a single instant of time, a response to the entire spectrum of electromagnetic waves. However, the selective approach to signal sweep into the frequency spectrum carries with it the problem of time delay, and this drawback can be eliminated only with difficulty. Thus, the above-described effect of time-unshifted reflections from the periodic structures cannot be attained. In comprehensive investigation of material structures for the micro-wave application (tensor and composite), the properties of materials are studied by means of classic single-frequency methods, which bring about certain difficulties in the research process [20]. In boundary changes with a size close to the wave-length, wrong information concerning the examined objects can occur. One of the possible ways of suppressing negative sources of signals consists in the use of wide-band signals such as white noise, and this approach can be further reinforced by analyzing the problem of absorption in the examined material. The indicated methods require a source of noise, a receiving and a transmitting antenna, and A/D conversion featuring a large bandwidth; for our purposes, the bandwidth ranged between 50 MHz and 10 GHz. Until recently, it had not been possible to design an A/D converter of the described speed or materialize devices with the above-mentioned bandwidth. Currently, high-end oscilloscopes are available with a sampling frequency of hundreds of Gsa/s.

2. NOISE SOURCE

At present, the appropriate type of source is supplied by certain manufacturers in the given field. Importantly, for the noise spectroscopy application, we require a comparatively large output power of up to 0 dB/mW; the assumed bandwidth characteristics then range up to 10 GHz. At this point, it is also necessary to mention the fundamental problem of finding active devices able to perform signal amplification at such high frequencies. Our requirements are thus limited by the current status of technology used in the production of commercially available devices; the highest-ranking solution for the bandwidth of up to 10 GHz can be found only up to the maximum of 0 dB/mW. In the noise spectroscopy experiments, we utilized a generator and an amplifier (NC1128A), Fig. 2. In order to verify the applicability of the noise spectroscopy laboratory arrangement (Fig. 4), we tested a metamaterial (periodic structure) designed for the frequency of 199.9 MHz (Fig. 3).

3. METHOD

Repeated transmission and sensing of both the signal provided by the noise generator and the external signals were carried out at the initial stage of the research. The repetition was performed for each sampled frequency, and the incident power spectrum was summed. Thus, we obtained the frequency dependence of the transmitted signal energy distribution. Generally, if the transmitter/receiver set is not located in a room with a defined spectral absorbance, we can expect uniform energy distribution within the whole frequency range. The record is, at its end, transformed to the frequency dependence of the specific power. In the described manner, we acquired the characteristics of the spectrum measurement background. At this point, the examined sample was placed

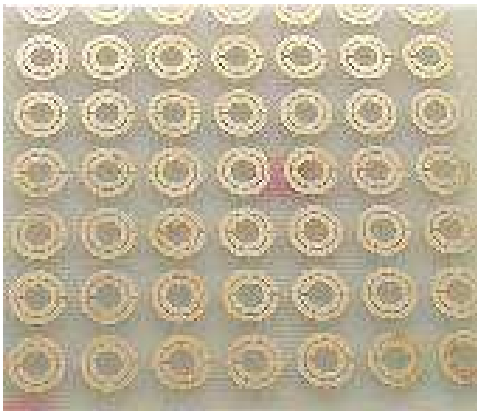


Figure 3: The first tuned periodic structure tested to verify the noise spectroscopy measurement.

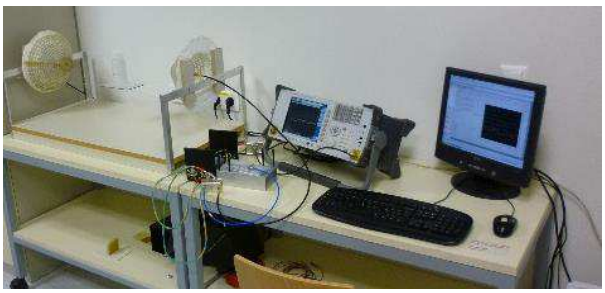
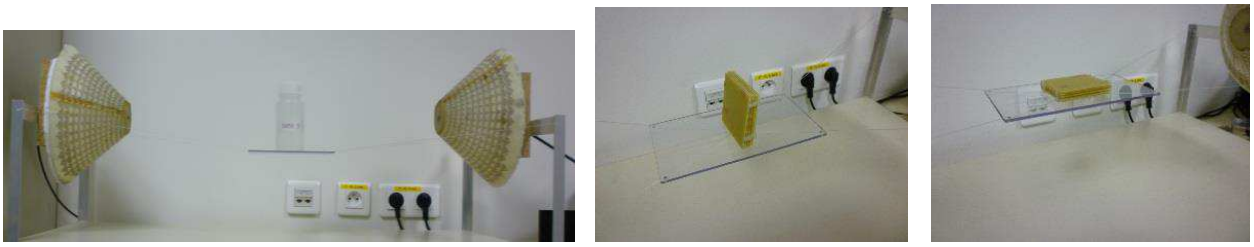


Figure 4: The arrangement of the noise spectroscopy station (free room).

in a support case (Figs. 4, 5(a), 5(b)); the sample for such application can be layered or periodic, and it is expected to provide the assumed frequency characteristic. Subsequently, repeated measurement was performed observing the above-outlined procedure. In the case of a markedly frequency-dependent background, the obtained characteristic can be corrected. Fig. 6 shows the frequency dependencies of the measurement setup and the multi-layer material.



(a) (b) (c)

Figure 5: (a) The liquid and (b) solid samples arranged in a laboratory for the sensing and evaluation of the spectrum (no shielded chamber; free room).

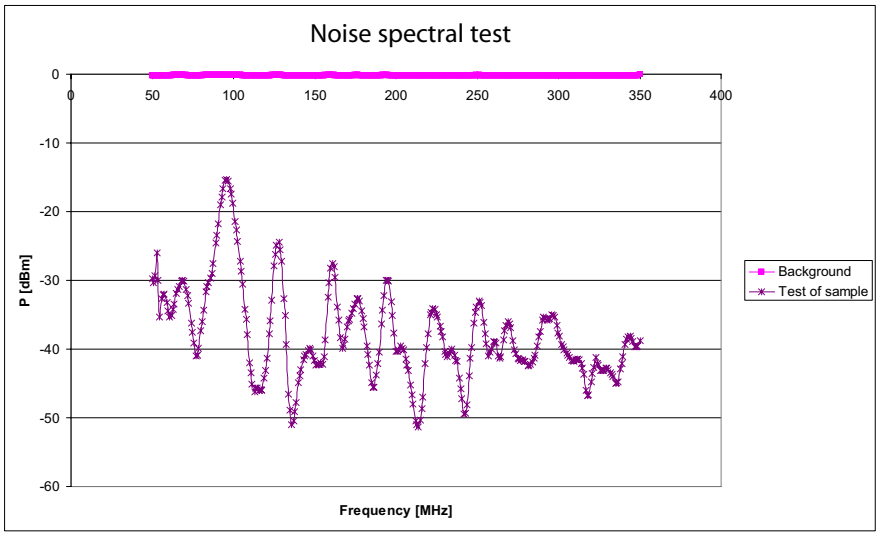


Figure 6: The waveform and spectrum of the noise generator output for the measured sample; $f = 50$–350 MHz.

4. CONCLUSION

The research paper provides an elementary overview and description of instrumentation for noise spectroscopy measurement and the related experiments. Noise spectroscopy operations within the frequency band of between 10 MHz and 10 GHz can be performed using currently available technologies. The noise source comprised a generator (NC1108A) and an amplifier (NC1128A).

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