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Non-Linear Electro-Ultrasonic Spectroscopy of resistive Materials

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Ústav fyziky

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NON-LINEAR ELECTRO-ULTRASONIC SPECTROSCOPY OF RESISTIVE MATERIALS

NELINEÁRNÍ ELEKTRO-ULTRAZVUKOVÁ SPEKTROSKOPIE REZISTORŮ

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INTRODUCTION

The non-destructive testing (NDT) has become an essential discipline for detection defects and cracks in the tested material without damage the sample. Therefore I can choose the samples which have not meet the standards. If a sample contains some defects, the important step is to localize the crack and define its properties (the crack's length, width, whether the crack will change its geometry in the future, etc.). The effort is to establish a lifetime of the sample or product, and separate the samples with short lifetimes. In this manner, I am able to eliminate fatal damage before the product fails. The non-destructive methods are of a great interest because the sample or product could be tested without its properties changing.

A lot of non-destructive testing methods are based on using of ultrasonic (ultrasound) signals. These methods help to analyze reflection, absorption and interference of mechanical wave. But at the same time these methods have some limitations. The tested sample must have a simple shape and made from a homogeneous material. Reflection of the ultrasonic wave from the edge of tested sample is a parasitic signal which can be evaluated as a defect or crack in the sample. The testing of the samples of small shape which are comparable with the ultrasonically induced mechanical wave length is very difficult. In this case ultrasonic signal of frequency over 10 MHz is used. But ultrasonic signal with higher frequency has higher absorption coefficient also. Therefore testing is performed on thin layers where the tested sample is situated in a liquid medium. The testing of the samples with un-homogenious structure and of difficult shape is impossible due to many parasitic signals.

There is a wide spectrum of non-destructive methods [1-4]. One of the new perspective methods is non-linear ultrasonic spectroscopy, which is useful for testing materials of complicated shapes, composite materials including connection feud and testing on the micro-structural level.

1 ELECTRO-ULTRASONIC SPECTROSCOPY

Electro-ultrasonic spectroscopy is closely related to electro-acoustic effects, which are known since 1933 [5]. These effects are resulted from a coupling between acoustic and electric fields. This phenomenon mostly occurs when ultrasound actuates on the fluid which contains electrically charged ions. Ultrasonic signal moves with ions and then this motion generates an AC electric signal. In general, it is a phenomenon, when mechanical wave influences electrical carriers directly. It occurs when mechanical wave has shorter wave length then mean free path of electrons. In other words, electric potential is measured due to mechanical wave on the material [6-8]. Audio devices are described by electro-acoustic effect also, where acoustic signal is converted to electric signal and conversely.

Electro-ultrasonic spectroscopy is based on interaction of ultrasonic signal and electric signal in conductive materials [9]. Ultrasonic signal changes the contact area between conducting grains in the sample, thus resistance of the sample is modulated by frequency of ultrasonic excitation. Defects and cracks in the sample structure are the sources of new intermodulation signal. The frequency of this signal is given by superposition or subtraction of exciting frequencies. This method is very sensitive because helps to evaluate the intermodulation signal on the frequency different from exciting frequencies of electrical and ultrasonic signal.

More information about electro-acoustic effect can be found in [10-11].

1.1 ULTRASONIC SIGNAL

Ultrasonic signal with frequency higher then 1 MHz is widely used in non-destructive testing. Electro-ultrasonic spectroscopy applies ultrasonic signal with frequency about 30 kHz. Thus the signal does not influence the electrical carriers directly. Standing waves are created on the sample by mechanical vibrations and sample’s geometry is changing in elastic range of deformations. Resistance of the sample is changing due to deformations, similarly as piezo-resistive effect. If the sample contains defects or cracks, then the resistance change is more significant.

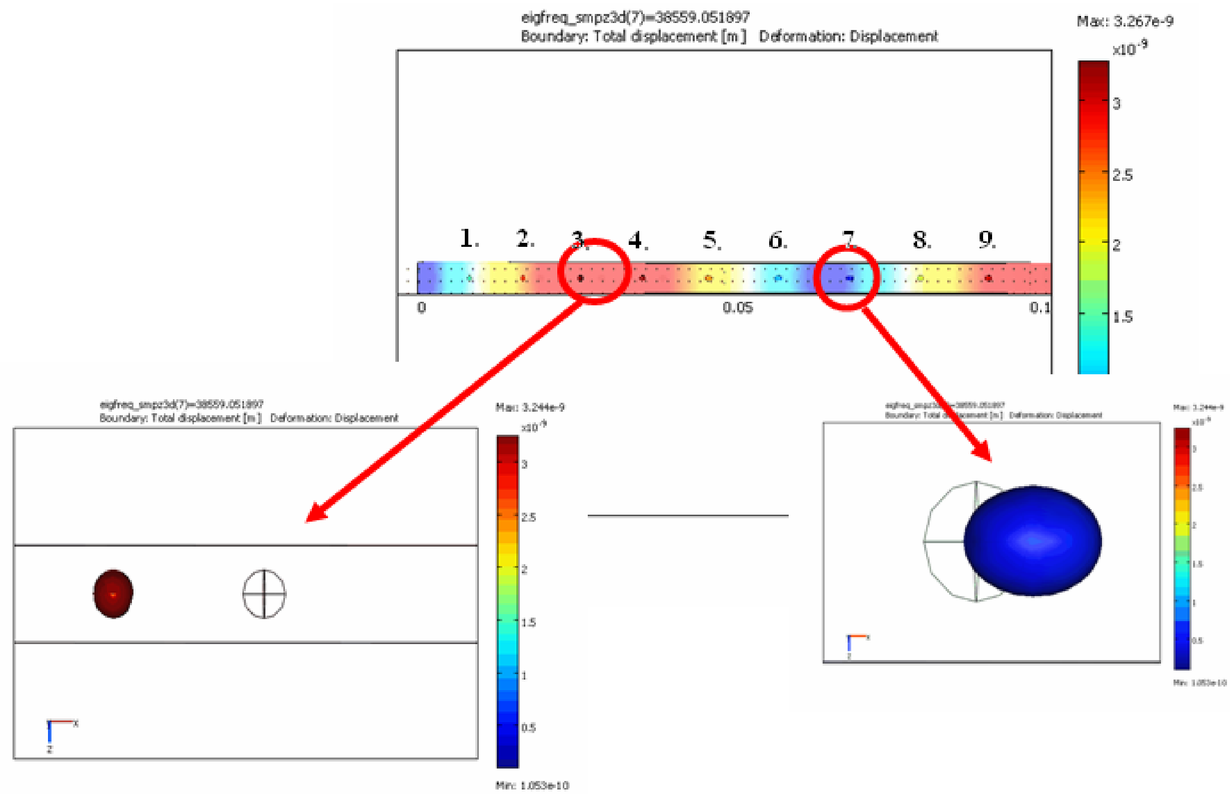

Fig. 1: *Longitudinal wave created on the metal sample*

Many types of standing waves may be created. Generally, it is longitudinal waves, transversal waves and torsion waves. Longitudinal wave is created on the metal sample, which has air bubbles inside the structure, see Fig. 1. Air bubbles are equally disposed from each other. This model was created in program COMSOL. In the area with minimal displacement the more volume's deformation occurs and vice verse. This helps to create a map of the sample's deformations and therefore deform any part of the sample. If the crack will be situated in minimal displacement area, then it will be more stressed, and resulting resistance change will be more significant.

1.2 ELECTRICAL SIGNAL

Generally, resistance change has very low value due to ultrasonically induced geometry change. Resistance change of homogeneous materials and sample without cracks is measurable also. It depends on the measurement setup and background noise.

Wheatstone bridge is the most sensitive method for measuring of a very low resistance change. If the electric current is flowing throw the sample and ultrasonic signal induces resistance change (Fig. 2), then the resulting electric spectrum is given by an equation (1):

$$V_{\mathrm{T}} = \left(R_{\mathrm{DUT}} + \Delta R\right) \cdot i_{\mathrm{AC}} \cos\left(\omega_{\mathrm{E}} t\right) = \left[R_{\mathrm{DUT}} + \Delta R \cos\left(\omega_{\mathrm{U}} t\right)\right] \cdot i_{\mathrm{AC}} \cos\left(\omega_{\mathrm{E}} t\right) = R_{\mathrm{DUT}} \cdot i_{\mathrm{AC}} \cos\left(\omega_{\mathrm{E}} t\right) + \frac{1}{2} \Delta R \cdot i_{\mathrm{AC}} \left[\cos\left(\omega_{\mathrm{E}} t - \omega_{\mathrm{U}} t\right) + \cos\left(\omega_{\mathrm{E}} t + \omega_{\mathrm{U}} t\right)\right] \tag{1}$$

where: R_{DUT} is resistance of the sample, i_{AC} is amplitude of electric current, ΔR is resistance change, ω_{E} and ω_{U} are angular frequencies.

Mechanical vibrations created by ultrasonic actuator induce a resistance change ΔR of the sample, with frequency f_{U}.

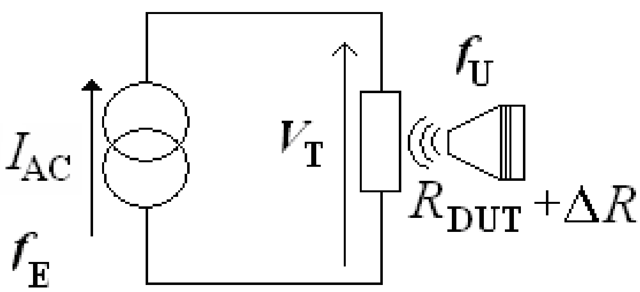

Fig. 2: *Electric circuit and ultrasonic transducer which influence the sample resistance*

The resultant voltage spectrum is given by voltage at frequency f_{E} which consists of resistance of the sample R_{DUT} and AC current flowing through the sample.

Sideband voltages are created by resistance change ΔR and AC current flowing through the sample structure also. The resultant spectrum is shown in Fig. 3.

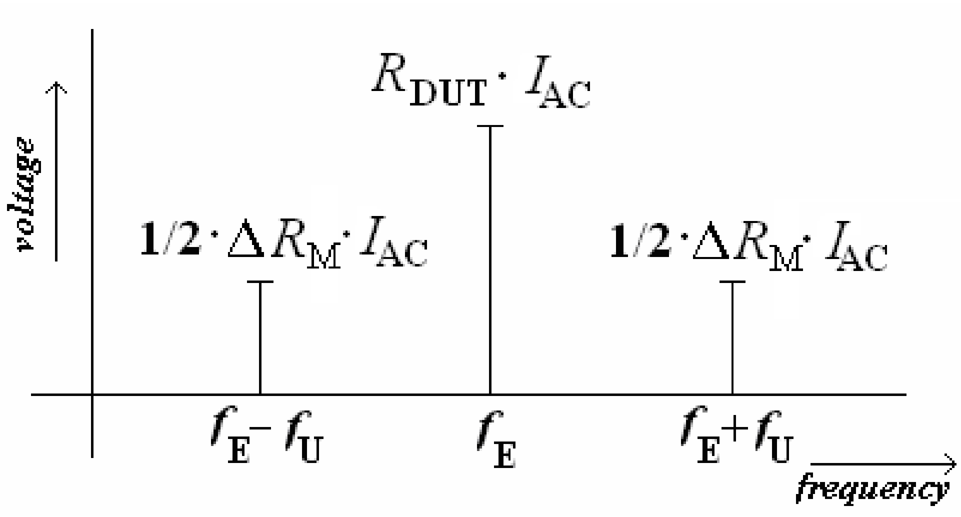

Fig. 3: *The theoretic resultant spectrum of the electro-ultrasonic spectroscopy*

The most sensitive method for measuring resistance, capacitance or inductance is connected to a Wheatstone bridge which basic circuit is shown in Fig. 4. Voltage measured between two arms of the Wheatstone bridge is given by (2):

$$V_x = \left[\frac{R_d}{R_c + R_d} - \frac{R_b}{R_a + R_b} \right] V_{IN} , \qquad (2)$$

The voltage V_x is zero, when the circuit is balanced and it is provided for values of resistors $R_d\, R_a = R_c\, R_b$.

For the balanced bridge the signal on the frequency f_E achieves minimal amplitude and should be a zero but due to parasitical inductances and capacities in the circuit it is not. When the circuit is balanced, then amplitude of sidebands voltage is given by the resistance change and electric current in arms of circuit with sample.

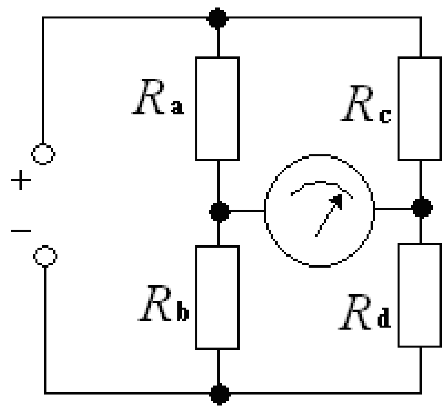

Fig. 4: *Resistors connected to the Wheatstone bridge*

Resistance change due to ultrasonic excitation is measured also for the sample without defects. It depends on the measurement setup. Noise background from these settings must be as low as possible compare with the signal on the intermodulation frequency.

Electric current which is used for measuring of resistance change, can be AC or DC. If DC electric current is used, then resistance change is measured on frequency of ultrasonic excitation. Advantage of AC electric current is possibility to measure resistance change on intermodulation frequency. Signal which gives information about the tested sample has frequency different from the exciting signals. In this case, it has high sensitivity.

2 ULTRASONIC ACTUATORS

The measurement was generally performed on an ultrasonic actuator HTP05. Actuators consist of two rings of PZT ceramics, copper contacts and metal body. The model of ultrasonic actuator HTP05 is shown in Fig. 5.

If AC voltage with frequency f_U from the electric generator is led to the copper contacts then mechanical vibrations are generated by the ultrasonic actuator variable with frequency f_U also. Advantages of these actuators are free operation maintenance and easy usage. On the other hand, disadvantage is the fact, that mechanical vibrations of the PZT ceramics are strongly dependent on the temperature and excited frequency from the generator.

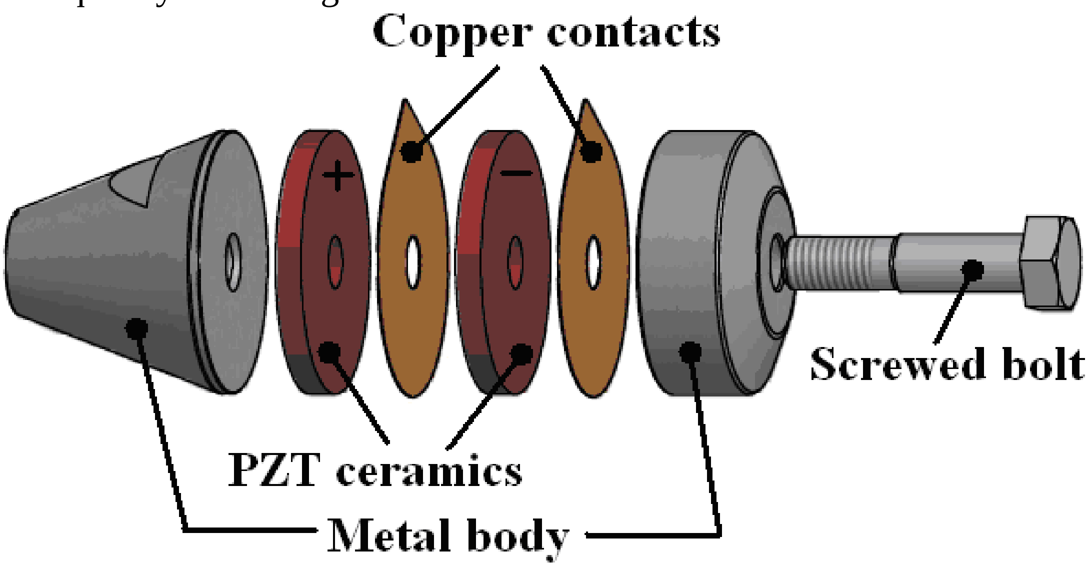

Fig. 5: *Model of the ultrasonic actuator HTP 05*

Displacement of mechanical oscillation has linear dependence on voltage led to the ultrasonic actuator from electric AC generator. Displacement of mechanical oscillation on the ultrasonic actuator HTP05 vs. amplitude of voltage V_U at different frequencies f_U = 25 kHz, 30.6 kHz and 32 kHz is shown in Fig. 6. The frequency

f_U = 30.6 kHz corresponds with resonant frequency of ultrasonic actuator. For this frequency displacement of mechanical oscillation has maximal amplitude.

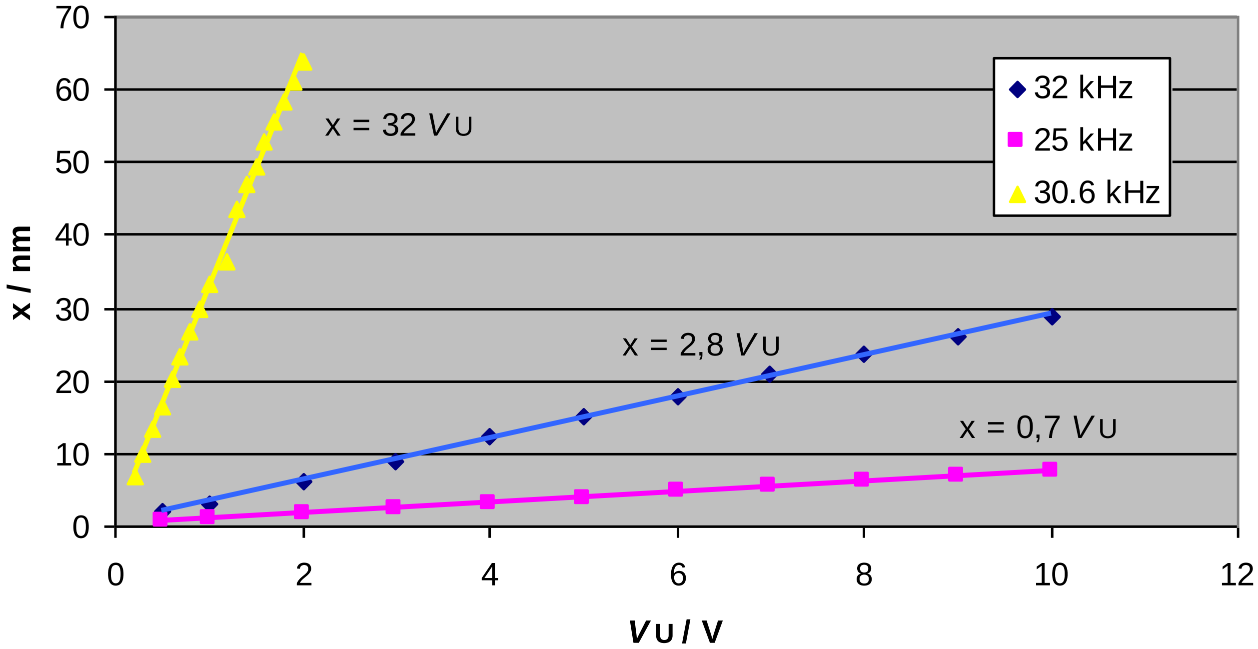

Fig. 6: *Displacement of mechanical oscillation x for ultrasonic actuator HTP05 vs. amplitude of voltage from AC generator with constant amplitude* V_U *= 5V for different frequencies* f_U *= 25 kHz, 30.6 kHz and 32 kHz*

The samples were fixed on the ultrasonic actuator by beeswax. Quality of the contacts is different for each sample between ultrasonic actuator and sample. Mechanical oscillations were controlled by sensor of acoustic emission on the sample.

3 AIMS OF DISSERTATION

The principal aim of this dissertation is to study a new non-destructive method to test resistors. This method is based on the interaction between the ultrasonic wave and the AC electric current. Ultrasonic phonons have an influence on transfer electrons located near defects. The aim is to find out the experimental information about the influence of ultrasonic phonons on the mobility of electrons. Due to different physical origins of ultrasonic and electric signals the electro-ultrasonic spectroscopy is supposed to provide higher resolution sensitivity for measured samples. The project is based on works of grants such as Non-linear ultrasonic spectroscopy in solids and Increasing the dynamic range of the analog signal pre-treatment (Nelineární ultrazvuková spektroskopie v pevných látkách and Zvyšování dynamického rozsahu systému s analogovým předzpracováním signálu).

3.1 REASON FOR ELECTRO-ULTRASONIC SPECTROSCOPY

Nowadays special attention is given to non-destructive methods of quality testing and the reliability of electronic devices. These methods keep the original parameters of the sample under the test. The electro-ultrasonic spectroscopy is a new method based on the interaction of two signals, the ultrasonic signal and the AC electrical signal. The high resolution of the electro-ultrasonic method is expected due to the fact that the measured signal is on the intermodulation frequency which differs from exciting frequencies.

3.2 PRINCIPAL AIMS ARE:

- Experimental study of the dependence of amplitude spectral density on the intermodulation frequency vs. amplitude of electrical AC voltage for constant ultrasonic excitation
- Experimental study of the dependence of the amplitude spectral density on the intermodulation frequency vs. the amplitude of the ultrasonic excitation for the constant electrical AC voltage
- Study the influence of the ultrasonic signal on both the sample structure and the resistance change which is induced by the ultrasonic signal
- Establish the influence of ultrasonic phonons on the mobility of electrons
- Measure many samples by means of electro-ultrasonic spectroscopy

4 EXPERIMENT

I measured various samples by means of the electro-ultrasonic spectroscopy. For example, samples of thick film resistors, metal samples of magnesium alloy, aluminium and dural plates, monocrystals Si and CdTe, varistors and granite samples. The intermodulation voltage was measured on a low frequency $f_i = f_E - f_U$. I studied the dependence of the intermodulation voltage on the amplitude of the electric current flowing through the tested sample and the amplitude of the ultrasonic excited resistance change.

I found out that the amplitude of the intermodulation voltage V_i is the linear function of the electrical excitation for constant ultrasonic excitation. It was observed for all measured samples.

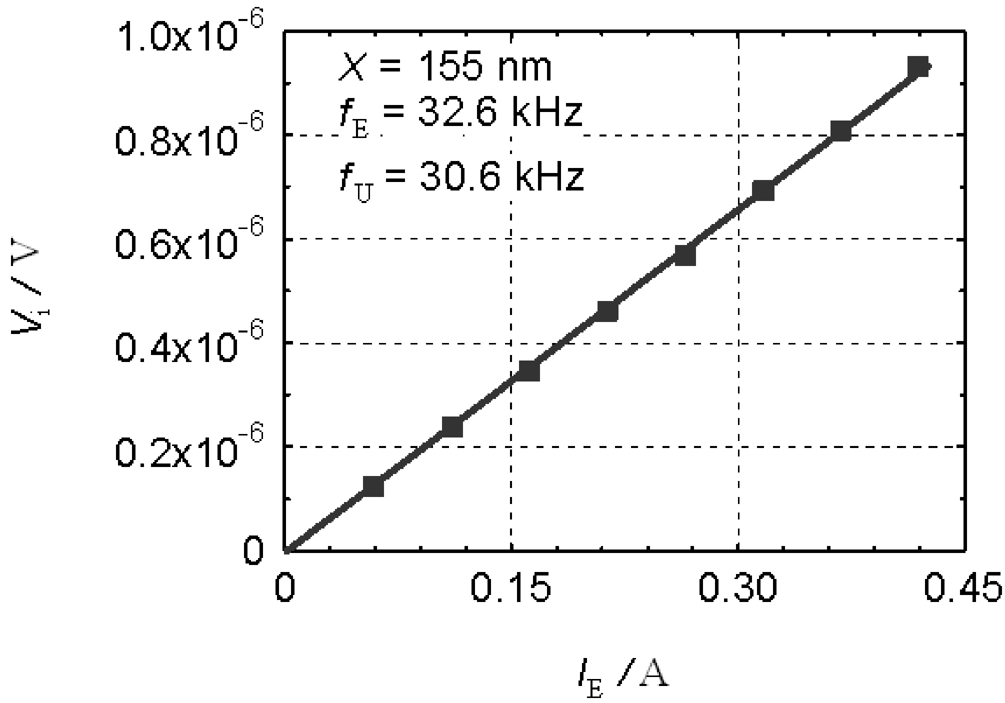

Fig. 7: *The voltage V_i on intermodulation frequency f_i vs. AC electric current I_E, for the constant amplitude of the sample dilatation X = 155 nm at frequency f_U = 30.6 kHz*

The dependence of the voltage V_{i} measured on the intermodulation frequency f_{i} on the AC electric current I_{E} is shown on Fig. 7. It was measured for the metal sample of magnesium alloy.

The amplitude of the intermodulation component depends on the ultrasonic excitation and several factors such as sample geometry, material, wave propagation etc. Fig. 8 shows changes in slope of the dependence of the intermodulation voltage on the ultrasonic excitation.

For most samples the ultrasonic excitation causes linearly increase of the intermodulation voltage. It works for longitudinal ultrasonic standing wave that is generated on the sample. It occurs if the direction of the electrical current is parallel to the direction of the ultrasonic excitation. When the ultrasonic excitation spreads perpendicular to the electrical current the different behavior of intermodulation voltage might be revealed. It was observed for metal samples where a metal plate was excited in longitudinal and transversal wave directions. The gauge factor is influenced by cracks, defects and inhomogeneities of the material. In other words the sample with defects is more sensitive on the ultrasonic excitation then the sample without defects. If samples are excited by the same ultrasonic wave the dependent of the intermodulation voltage on the ultrasonic excitation remains the same.

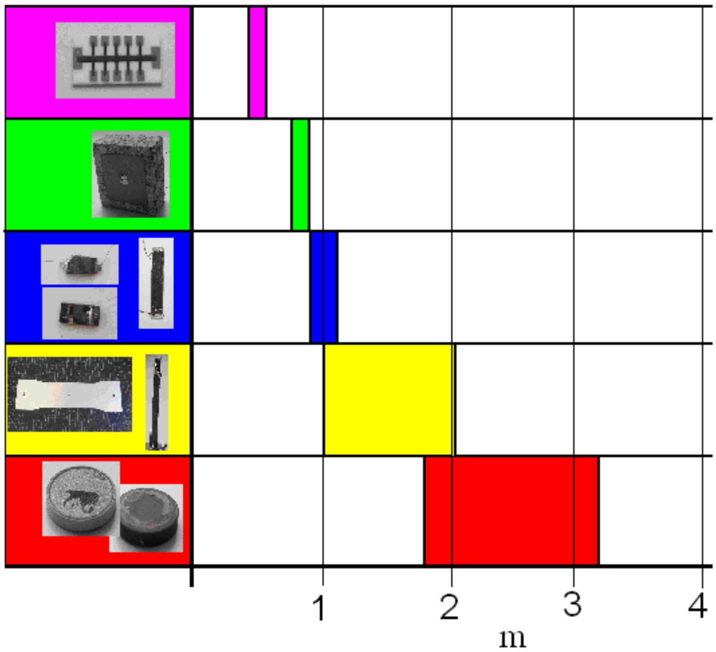

Fig. 8: *The slope of dependence of intermodulation voltage on ultrasonic excitation*

The types of the deformation were modeled by means of the program COMSOL. Torsion wave had the highest deformations inside structure of the sample, then there is longitudinal wave or transversal one.

Intermodulation voltage vs. damage of the sample was measured for granite sample. The specimen of granite was measured by means of the electro-ultrasonic spectroscopy. Then mechanical load (provided by hydraulic press) was applied on this sample.

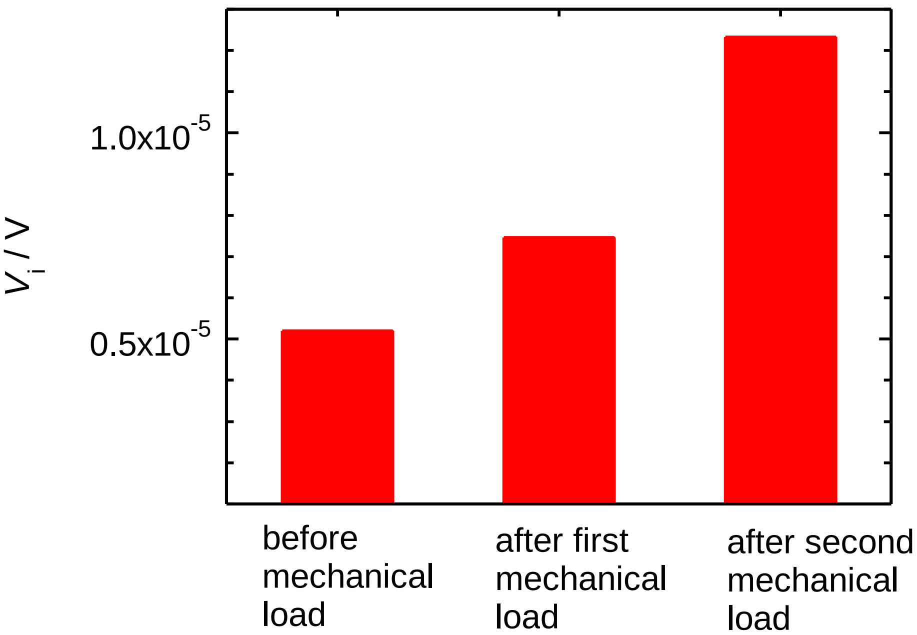

Fig. 9: *Intermodulation voltage V_i increasing with number of cracks in the granite sample*

Generally, an application of mechanical stress leads to the formation of micro-cracks in stressed solid dielectric materials. Cracks generation is accompanied by the generation of the electromagnetic (EME) and acoustic (AE) emission signals, which can be measured by appropriate sensors. Continual measurement and real-time processing and evaluation of these signals can be used for quantitative sample damage estimation. Then the mechanical load was relieved and the sample measuring was conducted one more time by means of the electro-ultrasonic spectroscopy. The dependence of voltage V_i (measured on intermodulation frequency f_i) on the granite sample damage is shown in Fig. 9. That reveals the increase of the intermodulation voltage V_i due to cracks into the sample structure.

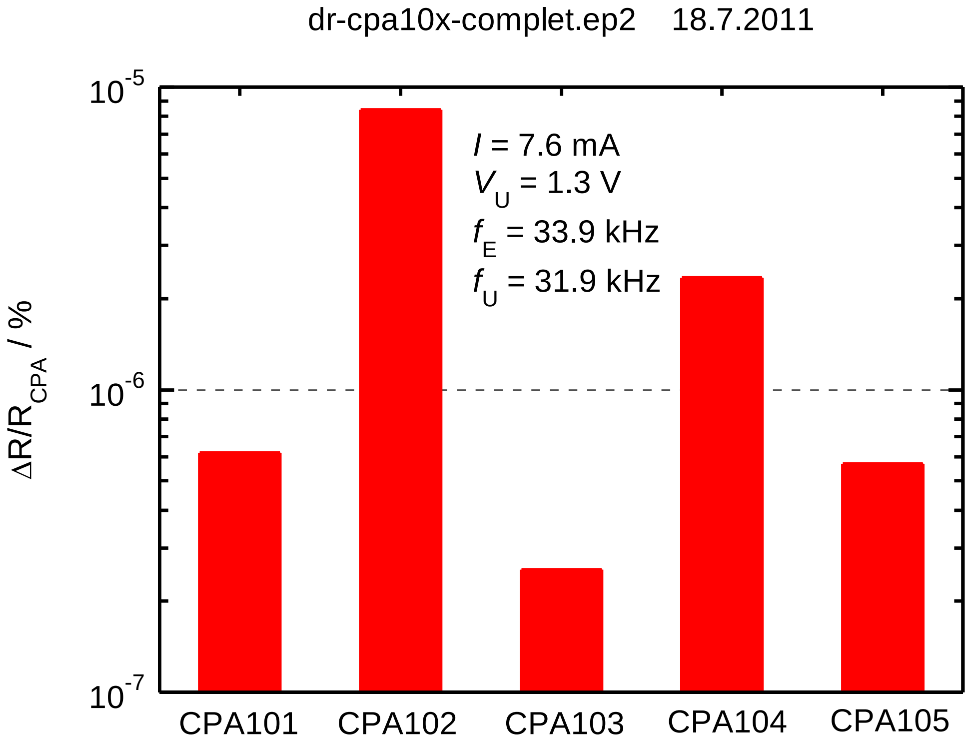

Fig. 10: *Relative resistance change for all measured samples, for AC current* $I_E = 7.6\ mA$

The amplitude of the induced resistance change caused by ultrasonic vibrations is very low. Fig. 10 shows the relative resistance change measured for five samples of thick film resistors. I found out that the relative resistance change $\Delta R/R_X$ is around of the order of 10^{-6} percent.

If a set of samples is measured the sample which has a higher influence on the ultrasonic vibration could be chosen. This sample contains some defects in volume structure evidently. On the other hand, the sample with a low influence on the ultrasonic vibration has less amount of defects in structure. In this illustrative case, sample CPA103 has the lowest sensitivity on the ultrasonic excitation.

5 CONTRIBUTION OF DOCTORAL THESIS

The method of the nondestructive measurement has never been described before and our department is the first one to do it. To provide nondestructive tests the ultrasonic signal is used. But quite different method is applied here. In case of electro-ultrasonic spectroscopy, the ultrasonic signal of low frequency is used only to generate standing mechanical oscillations in specimen. It causes the deformation of the specimen in the elastic range and contained structural defects could be revealed. The more structural defects the specimen has the more sensitive its reaction to the ultrasound signal is. Moreover, it could be used to examine the influence of mechanical oscillations on different electronic components. Each mechanical system is loaded with oscillations of different intensity. Damaged component or material could have different properties under the influence of these oscillations. It could cause fatal consequences. The method of electro ultrasonic spectroscopy could prevent these situations. By means of this method materials, components or the whole circuits which don’t comply with required properties under generated oscillations could be found out. This dissertation demonstrates practical measurements by means of electro ultrasonic spectroscopy for both conductive materials and semi-conductive ones or materials with high resistance.

6 CONCLUSION AND DISCUSSION

The method of electro-ultrasonic spectroscopy is a non-destructive method of testing. This method is based on the interaction between two signals: the electric AC signal and the ultrasonic signal. A new harmonic signal of the frequency f_i is created as a result of resistance change due to variation of the crack effective area by ultrasonic excitation.

The intermodulation frequency f_i is given by the subtraction of excitation frequencies f_E and f_U. The amplitude of the intermodulation signal on frequency f_i is influenced by the electric current flowing through the sample structure and ultrasonically induced resistance change due to the defects and inhomogeneities in the sample structure. High sensitivity of this method could be explained by the fact that the signal that gives information on tested sample has the frequency which differs from the exciting signals.

All materials contain cracks and micro-cracks in their structure. My aim is to detect these cracks. The Electro-Ultrasonic spectroscopy is a non–destructive method of testing which describes both the quality and the reliability of the tested sample.

I found out that the amplitude of the intermodulation voltage V_i is the linear function of the electrical excitation for the constant ultrasonic excitation. It was observed for all measured samples.

The amplitude of the intermodulation component depends on the ultrasonic excitation and sample geometry, material, wave propagation etc.

The ultrasonic signal is applied on the sample in a frequency range from 15 kHz up to 150 kHz. The wavelength of the ultrasonic waves is smaller then mean free path of carriers, thus, the ultrasonic wave does not influence the carriers directly. Ultrasonic excitation changes the geometry of the sample only, in the range of elastic deformations (the non-destructive geometry change). Thus standing wave is created on the sample by ultrasonic vibration. Resistance of the sample is changing similarly to piezoresistive effect. The sample structure is changing in its all volume due to this excitation. The geometry change inside the sample depends on the type of wave (longitudinal, transversal or torsion wave), frequency of oscillations and position where is defect situated inside the sample. Orientation of cracks considering is also important the wave propagation.

I measured various types of samples, such as thick film resistors, metal samples of magnesium alloy, aluminium and dural plates, monocrystals Si and CdTe, varistors and granite samples. Experimental results of measurements on the various samples by method of electro-ultrasonic spectroscopy were described in many papers for example [12-23].

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Languages

English

Abstract

The Electro-Ultrasonic spectroscopy is non–destructive method of testing which describes both the quality and the reliability of the tested sample. Tested sample is excited by the harmonic electrical signal of frequency f_E and the ultrasonic signal of frequency f_U. A new harmonic signal of the frequency f_i is created as a result of the electrical resistance change due to the variation of the crack effective area by ultrasonic excitation. The intermodulation frequency f_i is given by the subtraction of excitation frequencies f_E and f_U. The amplitude of the intermodulation signal on frequency f_i is influenced by the electric current flowing through the sample structure and the ultrasonically induced resistance change due to the defects and unhomogeneities in the sample structure. High sensitivity of this method follows from the fact that the signal that gives information on tested sample has the frequency which differs from the exciting signals. The “signal to noise” ratio and the high sensitivity for NDT analyses are based on the application of special electrical filters for attenuation of exciting signals in signal preprocessing. This work presents a new non-destructive testing method of solids with metallic electrical conductivity, monocrystals, resistive materials and electronic devices.

Abstrakt

Elektro-ultrazvuková spektroskopie je založena na interakci dvou signálů, elektrického střídavého signálu s frekvencí f_E a ultrazvukového signálu s frekvencí f_U. Ultrazvukový signál mění vzdálenost mezi vodivými zrny ve vzorku a tím mění jeho celkový elektrický odpor R. Změna odporu ΔR je proměnná s frekvencí ultrazvukového signálu f_U. Vzorek, který obsahuje mnoho defektů ve své struktuře, vykazuje vysokou změnu odporu ΔR v porovnání se vzorkem bez defektů při stejné hodnotě ultrazvukového a elektrického signálu. V disertační práci je popsána elektro-ultrazvuková metoda na tlustovrstvých rezistorech, hořčíkových slitinách, monokrystalech Si a CdTe, varistorech a také jeden z prvních pokusů aplikace elektro-ultrazvukové spektroskopie na horninové vzorky a tak diagnostikovat jejich stav poškození. V našem případě byl proměřen vzorek žuly. Jelikož se jedná o nedestruktivní metodu testování, tak má tato metoda velmi perspektivní budoucnost. Tato metoda je citlivá na všechny defekty ve vzorku. Její výhodou je, že se měří velikost signálu ne frekvenci danou rozdílem nebo součtem budících frekvencí f_E a f_U a tím se dá dosáhnout vysoké citlivosti. V mém případě byl vždy měřen signál na rozdílové frekvenci $f_m = f_E - f_U$.