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False Piezoresistive Effect Detection

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***Abstract*—Possibility of the use commercial carbon-polymer pastes for preparation thick-film resistors for linear strain sensors application was tested. The research showed that the polymer thick-film resistors are characterized by the piezoresistive effect up to 2% strain. For higher strain small cracks appeared in the resistive film. Presence of the cracks influenced resistance of the sensor. It is so called false piezoresistive effect. The cracks may cause instability of the sensor. Visual detection of the cracks is hardly possible because of their small dimensions. The indicator of cracks creation in resistive film was found.**

***Keywords*—polymer thick-film resistors; false piezoresistive effect; strain sensors**

I. Introduction

DYNAMIC development of Internet of Things and technological development of printed electronics determine using new combinations of different materials and technologies to achieve more functional devices. It is especially focused on new sensor constructions. One of the most popular solutions are extensometers (strain gauges) in which mechanical deformation causes a change of their electrical resistance. For these applications thick-film resistors made of carbon-polymer materials, such as resistive pastes, seem to be very promising. Due to the piezoresistive effect of these resistors it is possible to measure many physical quantities, such as load, deformation, acceleration, etc. in a moderate precise and repeatable way. Many research works were carried out to develop effective polymer piezoresistive materials, choose proper substrates for them and apply them for construction of specific sensors. Carbon black, carbon nanotubes, graphene, nanowires, nanoparticles and their mixtures were used as conductive fillers and silicone elastomers, rubbers or thermoplastic elastomers were applied as substrates for piezoresistors for skin mountable applications [1].

Self-made polymer piezoresistive pastes with matrix of thermoplastics elastomer and filler of multi-walled carbon nanotubes were prepared and deposited on thermoplastic elastomer substrate. Piezoresistive properties of sensors were maintained up to 80% strain [2].

Similar pastes were applied on PET substrates to form strain sensors of a Wheatstone bridge form [3]. Various deposition techniques were applied. Sensors made by screen printing were the most sensitive.

Green approach was undertaken too. Water soluble polyvinyl alcohol (PVA) filled with multi-walled carbon nanotubes (MWCNT) was applied to prepare a conductive paste [4]. Piezoresistive structures were screen printed or spray printed on PVA substrate. Response was linear and gauge factor was up to 3. The tested piezoresistors showed creeping during fatigue test.

Various polymers (PVDF, SEBS, TPU) were applied as a matrix for piezoresistive pastes as well as substrates [5]. The sensors were tested in bending and compression modes. It was concluded that PVDF is proper for pressure applications and TPU or SEBS for stretchable sensors.

Carbon nanostructures containing clusters of aligned MWCNT with a high degree of entanglement as a conductive filler and polydimethylsiloxane (PDMS) as a matrix were applied to prepare a piezoresistive tape [6]. The structures reached strain with linear response up to 30 % and gauge factor up to 47 depending on a CNS content. Resistance creep during cyclic stretch/release tests was observed.

The piezoresistive tape consisting of carbon nanotubes and carbon black mixture filler and poly(styrene-ethylene-butylene-styrene) matrix was prepared and tested [7]. Gauge factor about 10 and sensor maximum elongation 20 % were reached. Hysteresis and creep took place during cyclic stretching.

Carbon nanotubes in a SU-8 photopolymer as a self-made piezoresistive material and polyethylene terephthalate (PET) were used to form piezoresistve structures [8]. The SU-8 photopolymer was applied to prepare cantilever MEMS structure.

Self-made pastes containing ellipsoidal graphite and epoxy resin were tested on alumina and aluminum substrates. Creep of resistance was observed [9].

Commercial polymer pastes and unconventional for electronic industry substrates of biomedical grade silicon rubber were applied for implantable thick-film strain sensor [10]. Gauge factor of 2.5 was reached. Maximum strain was about 2 % because of resistive film cracking.

Commercial resistive pastes and rigid epoxy-fiberglass printed circuit board substrates were used for load sensors for medical applications [11]. Drift of 5% of output signal was observed.

Commercial polymer resistive pastes were deposited on glass-epoxy FR4 substrate [12]. It was observed that piezoresistors are three times more sensitive to longitudinal strain than to transverse one. There was observed small

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resistance drift of 0.2% after exposure to strain.

The authors endeavoured to test commercially available resistive polymer paste and substrate as possible candidates for manufacturing cheap, repeatable, moderate precise and stable piezoresistive sensors for various consumer applications. The authors assume that the use of commercial materials can lead to quick implementation sensors to production.

II. METHODS

The investigations were realized with the use of thick-film resistors made of resistive paste with a sheet resistance of 1kΩ/□/25µm (MINICO M2013 RS MOD2 – carbon paste) from Acheson. Contact pads were made of conductive silver paste (Electrodag PF-050) from Acheson. The conductive (for contact pads) and resistive (for resistors) pastes were applied with the screen-printing method with the use of semiautomatic screen printer (ACCU-COAT 3230 from AREMCO PRODUCTS) through a yellow PET mesh (77T) screen covered with a capillary 25 µm film. The screen printing operation was done with velocity of squeegee movement about 30-50 mm/s, and 0.6-0.7 mm distance between mesh and substrate material. The squeegee was made of rubber „diamond" type with hardness equal to 80 (Shore scale) and 45° angle.

The thickness of the applied polymer film in liquid phase was approximately 25 µm. After printing of each paste the test samples were cured in a heat oven for 30 minutes at 180 °C. Flexible DuPont Polyimide Kapton® 500HN and Kapton® 400HN films were used as substrates. The layout of formed resistors is presented in Fig. 1. The relative short length of conductive tracks was made deliberately for independence of their electrical resistance changes during elongation tests.

Kapton 400HN Kapton 500HN

Fig. 1. The test samples for mechanical strain.

The resistors were made in configuration as a single square with 2.5 mm side, which are placed longitudinally (signed as RL) and transversally (signed as RT) to the line of strain.

The prepared test samples in the form of stripes, with applied resistors, were subjected to stretch tests. The tests were realized with the use of a tensile and compression test system (Force Testing Machine, Mecmesin Multitest i-1; 500N option) with force and elongation/shortening in real time mode measurements.

The electrical resistance of resistors during all the tests was measured with the use an Agilent 34401A laboratory multimeter in the real time measurements collection configuration mode.

Electrical tests were followed by microscope observation. Optical and scanning electron microscopes were applied to detect possible damage of tested resistors.

Optical microscope observations were carried out just at the tensile and compression test system. Samples for SEM observations were subjected to the strain, glued to steel blocks and placed in the SEM chamber.

III. RESULTS AND DISCUSSION

Figure 2 presents characteristics of electrical resistance changes of longitudinal (RL) and transversal (RT) resistors made on the Kapton® substrate material. During investigations the polyimide of 100 µm (signed as 400HN) and 125 µm (signed as 500HN) thickness was used. As it can be seen on the graphs, all characteristics may be divided into three subranges: A – the first range of nonlinear changes; B – the range of linear changes; C – the second range of nonlinear changes. Resistance changes in the subranges A and B were reversible. Resistance decreased to initial value after strain releasing.

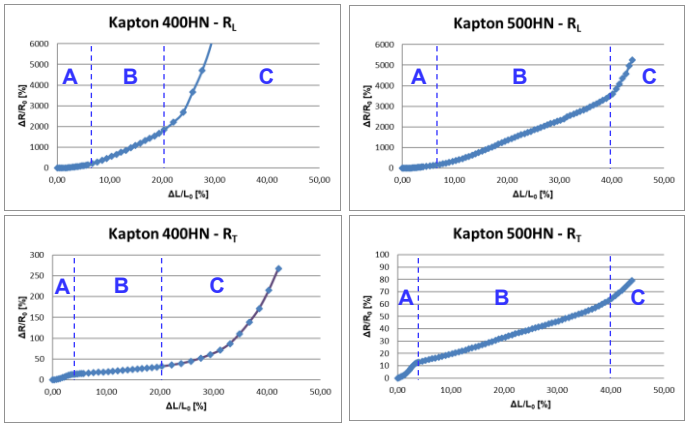


Fig. 2. Relative changes of electrical resistance during stretch tests for full range of tests.

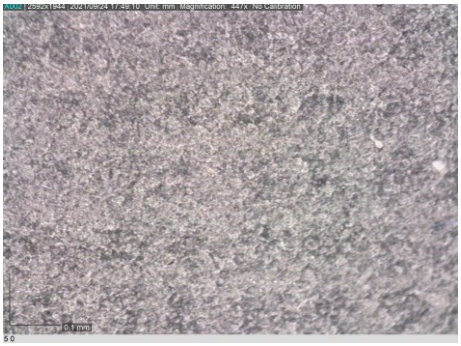
Similar character of resistance change was observed in [8]. The measurements were carried out up to strain of 3.5 %. Resistors containing less 2 % conductive filler (filler contents about percolation threshold) showed change of piezoresistive response at about 2 % strain. As a matter of fact in the relative variation of resistance as a function of applied strain presented in [8] three regions may be distinguished. At high strain the third region of very fast resistance growth for resistors of low carbon nanotubes concentration can be noticed.

Similar relative resistance versus tensile strain changes were reported in [13]. Damage of conducting network in the conductive film was suspected as the reason of such behaviour. Various values of elastic deformation of piezoresistors were reported 2 % [5] and 80% [2]. The range of elastic strain of piezoresistor is dependent on the polymer matrix material of resistor and the substrate material.

The end of linear range for Kapton 500HN polyimide material is almost two times higher than in case of the 400HN material. The reason is thicker substrate material that is more resistant to deformation (linear elongation) which causes its permanent damage.

Relative resistance changes of resistors on Kapton 500HN during stretch tests were followed by microscope observation of resistors under strain. Damage of resistive film was sought, as it was reported that polymer resistors may be damaged during strain [10], [13]. The cracks may appear in resistive film causing resistance growth. These cracks may disappear when strain is released. As a result the separated carbon segments return to contact and resistance decreases. This phenomenon may be faulty admitted as piezoresistivity. Similar effect was reported earlier for cermet resistors [14]. It was called false piezoresistive properties.

Microscope pictures of resistors under strain are presented in Figures 3 to 9.



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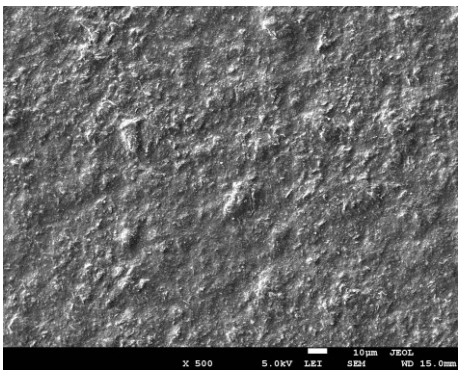
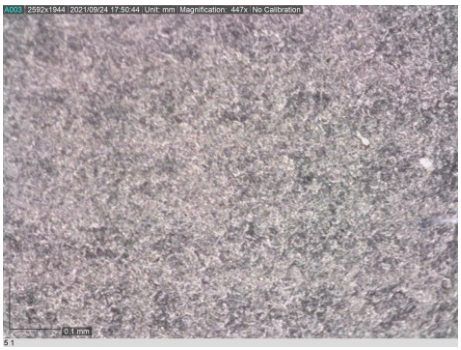


(b)

Fig. 3. The resistor without strain (a) optical microscope picture (b) SEM picture



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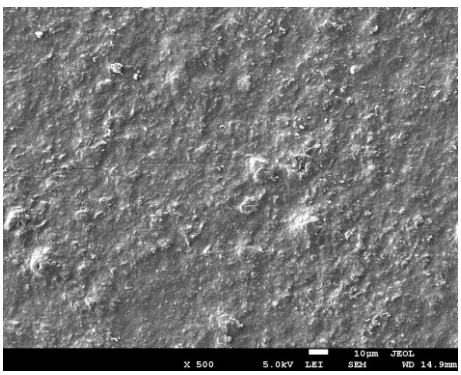

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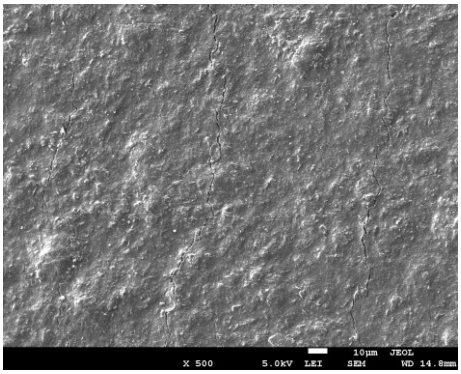
Fig. 4. The resistor under tensile strain of 1.25% (a) optical microscope picture (b) SEM picture

(a)

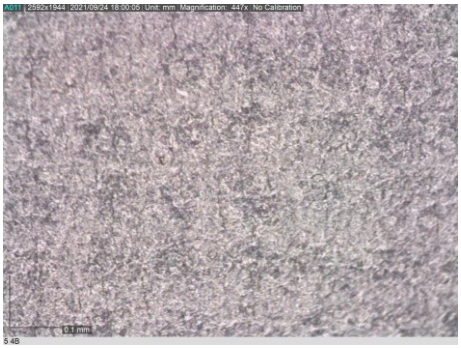


(b)

Fig. 5. The resistor under tensile strain of 2.5% (a) optical microscope picture (b) SEM picture

Resistor surface without strain is presented in Fig. 3. It may be seen that SEM picture is much more clear and more details may be noticed. No significant damage under strain of 1.25% is visible in Fig. 4. neither in optical nor in SEM picture. Under strain of 2,5% (Fig. 5) small cracks on resistor surface appeared. The cracks are placed perpendicularly to strain direction. The cracks are visible in SEM picture only. Under strain of 5% (Fig. 6) the cracks are hardly visible in the optical microscope picture too. No openings in resistive film can be noticed in the optical picture in the backlight. When strain was released the cracks disappeared in the optical microscope picture. Figure 7. presents the resistor under tensile strain of 7.5%. The cracks are clearly visible in the optical microscope picture. Some holes appeared in the resistive film that can be seen in the optical microscope picture in the backlight. The cracks in resistive film reached the substrate. Under tensile strain of 13,75% (Fig. 8) long cracks are visible in the optical picture in the backlight. When strain released the cracks remained visible in optical microscope picture. Under tensile strain of 30% the long and wide cracks perpendicular to strain direction were visible (Fig. 9).

The shape of the cracks is irregular. It is difficult to estimate density of the cracks and their width. Number and width of the cracks increased with strain. For strain value up to 5% the cracks did not penetrate resistive film up to the substrate. For strain over 7.5% the cracks broke through resistive layer completely but conductive bridges still remained and conductivity of the resistor was still sustained.



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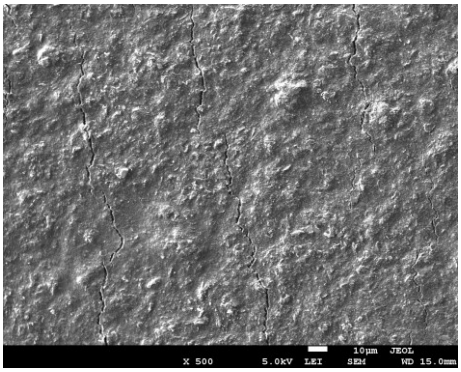

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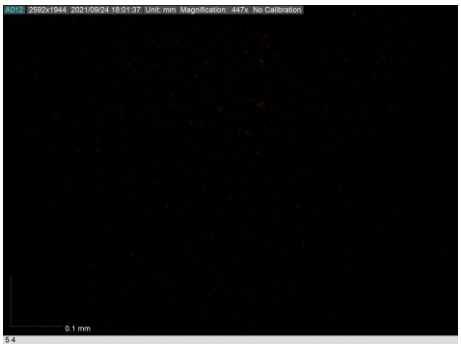

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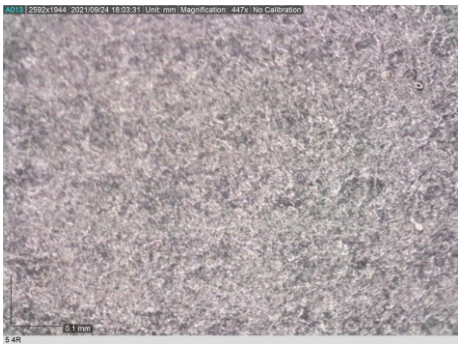
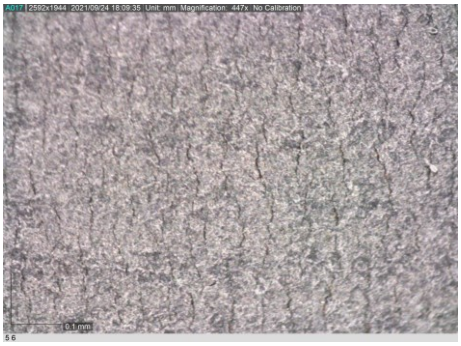


(d)

Fig. 6. The resistor under tensile strain of 5% (a) optical microscope picture (b) SEM picture (c) optical microscope picture in the backlight (d). optical microscope picture when strain released



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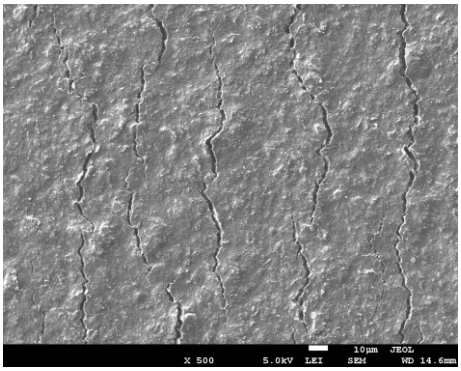

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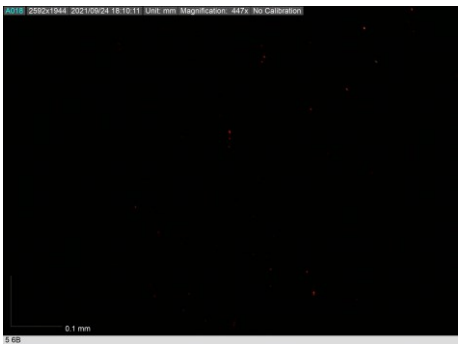

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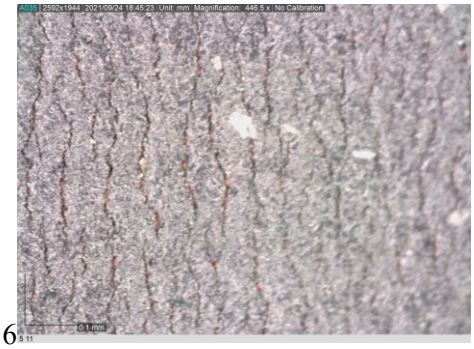
Fig. 7. The resistor under tensile strain of 7.5% (a) optical microscope picture (b) SEM picture (c) optical microscope picture in the backlight (d) optical microscope picture when strain released



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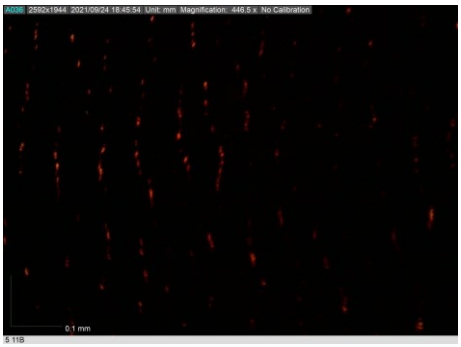


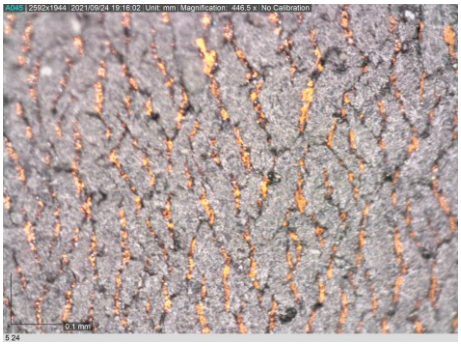
(b)

(c)

Fig. 8. The resistor under tensile strain of 13.75% (a) optical microscope picture (b) optical microscope picture in the backlight (c) optical microscope picture when strain released

Resistance changes caused by strain were reversible up to strain of 40%. However application such resistors as extensometers in such wide range seems to be risky. Behaviour of cracks in long term application may be unstable and unexpected. Such instability may be caused by moisture or contamination of crack sides. It is reasonable to limit application range of developed resistor structures to their piezoresistive behaviour range.

Figure 10 presents characteristics of electrical resistance changes of longitudinal (RL) resistor made on the polyimide Kapton® substrate material of 125 µm thickness (signed as 500HN) in the strain range from 0% to 2 %. Characteristic is not linear. Gauge factor may be estimated between 4 and 8. The results were repeatable for many samples and for many strain cycles. It seems that extensometer of elaborated construction may be successfully applied for moderate precise deformation measurements in this strain range.



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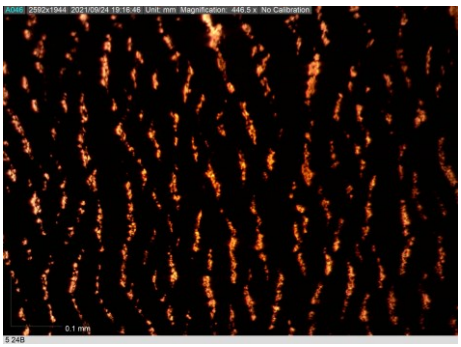

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Fig. 9. The resistor under tensile strain of 30% (a) optical microscope picture, (b) optical microscope picture in the backlight

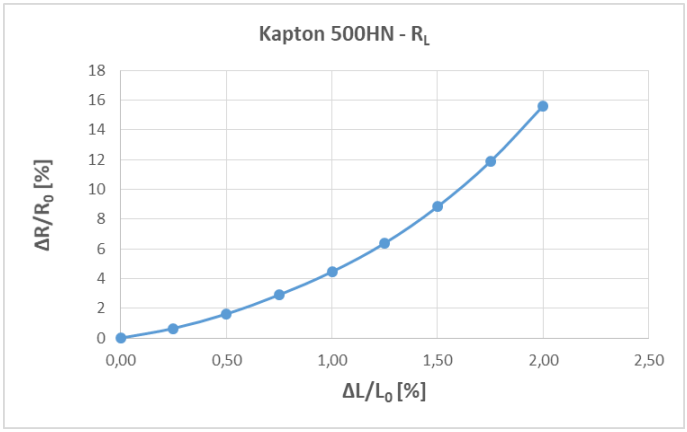

Fig. 10. Relative changes of electrical resistance during stretch tests in applicable strain range

The application limit of elaborated extensometer was estimated by observation of samples under strain with SEM. It was time consuming and expensive procedure. It seems that this limit could be estimated in the simpler way. The result of the cracks is resistance growth, just like result of stretching strain of piezoresistive material. The cracks that appear in the resistive film are placed perpendicularly to strain direction so longitudinal (to the strain) resistance growth caused by the cracks should be higher than transversal (to the strain) resistance growth.

Coefficient (ΔRL/RL0)/(ΔRT/RT0) as function of strain is presented in Figure 11.

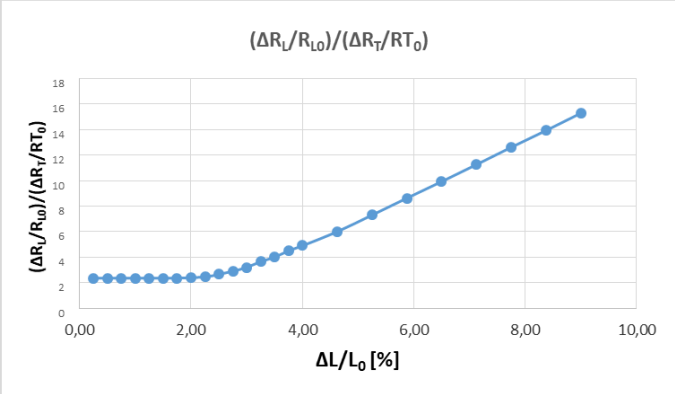

Fig. 11. Quotient of relative changes of longitudinal and transversal resistance during stretch tests.

Quotient (ΔRL/RL0)/(ΔRT/RT0) is stable up to about of 2% of strain. Coefficient (ΔRL/RL0)/(ΔRT/RT0) starts to grow about 2% strain. It is just the value of strain when the cracks in the resistive film appear and may be observed with SEM. It seems that (ΔRL/RL0)/(ΔRT/RT0) dependence of strain is a good indicator or cracks appearance in the resistive film.

CONCLUSIONS

Some commercial materials were tested as substrates for thick film carbon-polymer piezoresistors. It was found that the suitable choice of materials for both substrate material and resistive layer of resistor is crucial for constructing piezoresistive sensors. The uniform elongation of the substrate is necessary.

Kapton foil seems to be the best choice. It was possible to form piezoresistive sensors with repeatable response in range of elongation up to 2 %.

The indicator of cracks formation in the resistive film was found. It may be useful to estimate the maximum allowable limit of applied strain.

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