RIGA TECHNICAL UNIVERSITY

Faculty of Material Science and Applied Chemistry

Institute of Technical Physics

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PIEZORESISTIVE PROPERTIES OF ELASTOMER NANOGRAPHITE COMPOSITE SENSORELEMENT SYSTEMS

Summary of doctoral thesis

Scientific supervisor

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APPROVAL

I approve that I have elaborated the doctoral dissertation, which is submitted for consideration at Riga Technical Universtity for doctoral degree in physics. The doctoral dissertation has not been submitted at any other university for acquisition of a scientific degree.

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Date:

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CONTENT

GENERAL DISCRIPTION OF THE WORK

Introduction

Nowadays mechanical sensors are widely used in everyday life and in industry to control and monitor various processes. Industrial sensors are available in many kinds that substantially differ in design, accuracy class, cost, utilization conditions and detection principle. Most of the pressure sensors are made from solid materials like piezoresistive semiconductors, piezoelectric ceramics, magnetostrictive and capacitive metals or their alloys or oxides. Most of these materials are fragile to fast speed impact which can limit their use in applications of dynamic changes. Piezoresistive effect studies of various elastomer nanocarbon composites has been made for more than a decade at RTU Institute of Technical Physics. It was concluded that it is possible to obtain piezoresistive response during mechanical exposure of composites made in cetain condutions. Compared to previously mentioned sensors these are resistant to mechanical shocks and are able to detect impacts due to hyperelastic nature of composite matrix. These properties in combination with simple sensor manufacturing process, cheap raw materials and easy detection of sensor output makes them potentially attractive for new kind of sensor production.

Aim

Manufacture polyisoprene nanocarbon composites and evaluate different carbon allotrope and concentration influence on the piezoresistive effect. Determine the ambient temperature and mechanical loading frequency impact on the piezoresistive effect. Quantitatively describe composites electrical resistivity dependance of temperature and elaborate entirely hyperelastic pressure sensor systems in various sizes.

Tasks

1. To manufacture polyisoprene nanocarbon composites and determine:
    a) Various nanocarbon allotrope influence on piezoresistive sensitivity
    b) Ambient temperature influence on piezoresistive sensitivity
    c) Mechanical loading frequency influence on piezoresistive sensitivity
    d) composite electrical resistivity dependance of temperature
2. Mathematical modelling of composite electrical resistivity dependance of temperature using in literature mentioned models as well as elaboration of new mathematical model that discribes positive temperature coefficient of electrical resistivity
3. Elaboration of various size entirely hyperelastic pressure sensor systems

Scientific novelty

It is proved that piezoresistive effect in polyisoprene nanocarbon composites is temperature dependant. Conducting structure of composites determines the influence of temperature.

THESIS

1) Of all the tested polyisoprene various nanocarbon allotrope composites polyisoprene / thermally exfoliated graphite composite has the highest piezoresistive sensitivity due to the 2D filler mutual contact surface area reduction during crossdeformation leading to additional reduction of tunnelling currents.
2) The piezoresistive sensitivity increase with increase of ambient temperatures is due to reduction of polyisoprene tangent modulus therefore at the same mechanical stress larger deformations are achieved. Composites containing only one type of conducting filler therefore experience higher reduction of tunneling currents and consequent raise of electrical resistivity.
3) Increase of ambient temperature does not lead to statistically significant influence to piezoresistive sensitivity of PiCNTs(x)CB(y) composites whom main electrical structure are made of CNTs. This is due to simultaneous CB induced separation of individual CNTs from the electrically conductive channels and stimulation of new conductive channel formation.
4) Polyisoprene/nanographite and polycaprolactone/CNT composite positive temperature coefficient of electrical resistivity is explained with a mathematical model that is based on thermal expansion of the composite matrix induced reduction of tunelling currents and subsequent destruction of conductive channels.

1. REVIEW OF LITERATURE

In the literature review various production methods of electroconductive fillers are summarized. Production of natural caoutchouc as well as natural rubber based on various accelerated sulfur vulcanization systhems are reviewed. Also piezoresistive effect in various polymer conductive filler composites are analyzed based on used composite matrix and conductive fillers.

From the literature review it was concluded that one of the most possible application of piezoresistive polymer composites is to use them as cheap, variable size and shape, tactile sensors . Since integration of these tactile sensors into motorized devices would allow to achieve primitive tactile functions for detection of interactions with the environment. Similarly as tactile senses in living organisms provides necessary information to ensure body coordination and warning of potential dangers.

Analysis of piezoresistive sensitivity in polymer graphene composites were made. Wide distribution of the results were obtained, suggesting that sensitivity is strongly dependant on composite matrix, filler geometry and sensor production method.

2. EXPERIMENTAL PART

Experimental part is divided in to 3 subsections. At the first subsection dependance of piezoresistive sensitivity in polyisoprene nanographite composites is analyzed in terms of conductive filler influence - various carbon allotropes are evaluated. From these results (Fig. 1.) it was concluded that composites made from polyisoprene thermally exfoliated graphite has the highest piezoresistive sensitivity due to the 2D filler mutual contact surface area reduction during crossdeformation leading to additional reduction of tunnelling currents.

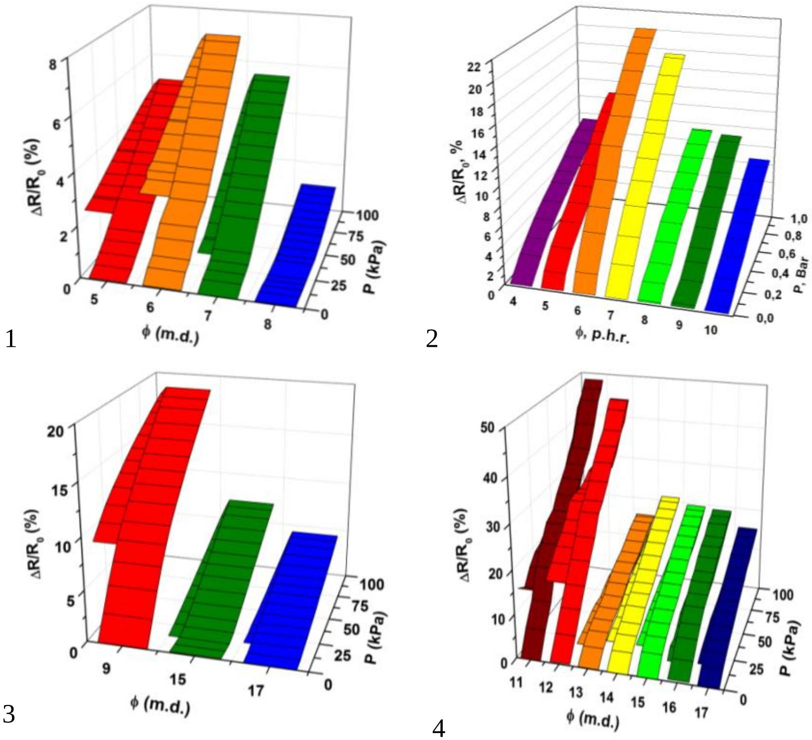


Fig. 1. Piezoresistive sensitivity of various carbon allotrope polyisoprene composites with different filler concentrations under single exposure of 100 kPa pressure. 1) PiCNT; 2)PiCB [1]; 3)PiCNTs; and 4)PiTEG composites

Based on these results polyisoprene composites containing two different electrically conductive fillers with diverse filler concentrations were made. These composites further in text is reffered as PiCNTs(x)CB(y) where „x" and „y" represents concentrations. The piezoresistive sensitivity dependance of these concentrations are shown in Fig. 2.

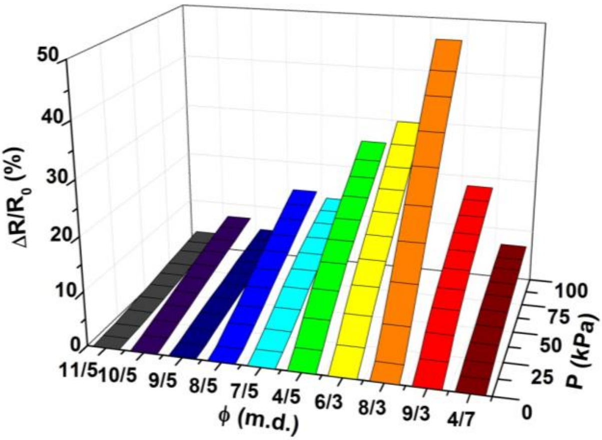


Fig. 2. Piezoresistive sensitivity of PiCNTs(x)CB(y) composites with different filler concentrations under single exposure of 100 kPa pressure

Compared to previous results shown in Fig. 1. as PiCNTs and PiCB one can see significant increase of sensitivity at specific PiCNTs(x)CB(y) concentrations. The increase in piezoresistive sensitivity could be explained as follows: Conductivity in PiCNT(x)CB(y) are formed from CNT and CB clusters where CB binds the unconnected CNT together to form conductive channels. During mechanical deformation, filler slippage occurs due to significant shear forces, but since the CB is a 0 D nanomaterial it should exhibit better mobility in composite microstructure compared to 1D CNT. This leads to a more complete conductive grid breakdown in equal uni-axial deformations in case of hybrid composites compared to PiCNT and PiCB composites where more significant shunting of channels might occur besides the breakdown of conductive channels because the mobilities of conductive particles are comparable. All this leads to an idea that the maximal PiCNT(x)CB(y) sensitivity can be achieved when the CB concentration is adequate to ensure the synergy between CNT and CB particles in formation of conductive grid. Additional advantage for these composites is partial replacement of expensive filler (CNTs) with cheaper (CB).

In the second subsection impact of ambient temperature on composite sensitivity is evaluated in accordance with ASTM D 1349 – 99 standard. This standard specifies temperatures at which properties of rubbers should be determined. By taking into account all possible resources piezoresistive effects of polyisoprene nanographite composites (PNCC) were determined at 20 °C, 40 °C and 55 °C

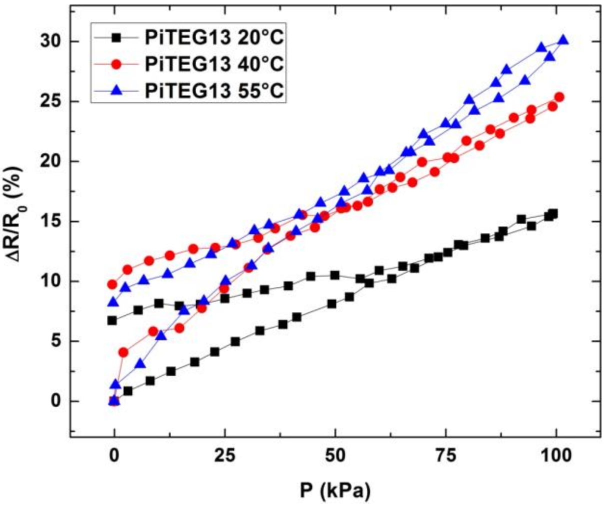


Fig. 3. Piezoresistive sensitivity of PiTEG 13 composite under single exposure of 100 kPa pressure at various ambient temperatures

The piezoresistive sensitivity of all PiTEG composites increases as shown in Fig. 3. This is explained as fallows: The piezoresistive sensitivity increases with increase of ambient temperatures due to reduction of polyisoprene tangent modulus therefore at the same mechanical stress larger deformations are achieved. Therefore these composites experience higher reduction of tunneling currents and consequent raise of electrical resistivity.

This statement was confirmed in tensile mode at various ambient temperatures where required force to deform composite sample (70x10x1mm) by 10% normal strain was messured. From Fig. 4. one can clearly see that the force required to achieve 10% normal strain is lower with increase of temperature.

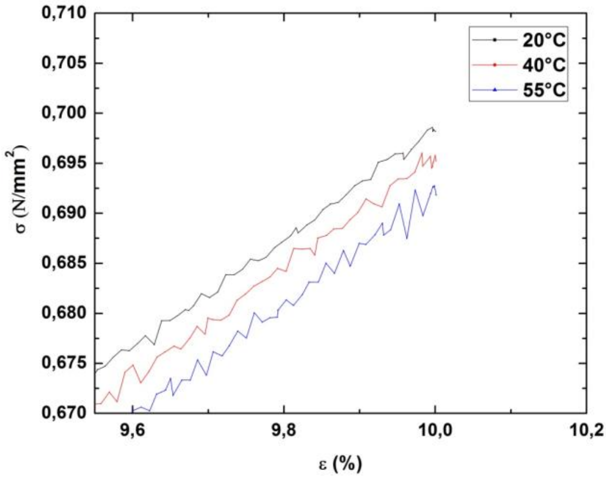


Fig. 4. Stress – strain curve of PNCC at various ambient temperatures

However piezoresistive sensitivity increase with increase of temperature as in PiTEG composites was not so exact in PiCNTs(x)CB(y). Even better some compositions like PiCNTs(4)CB(5) showed decrease of sensitivity. Besides that PiCNTs(10)CB(5) showed no statistically significant influence to piezoresistive sensitivity with increase of ambient temperature as shown in Fig. 5.

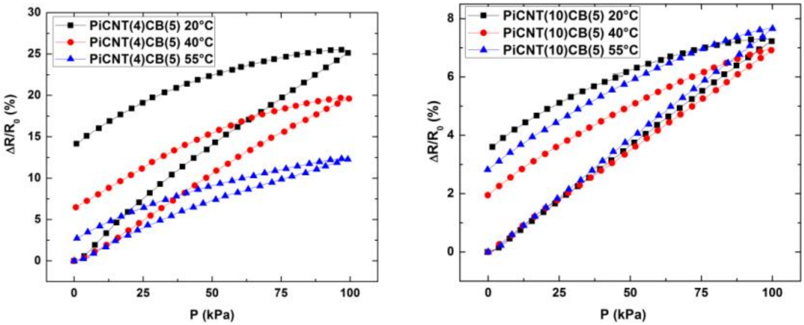


Fig. 5. Piezoresistive sensitivity of PiCNTs(4)CB(5) and PiCNTs(10)CB(5) composites under single exposure of 100 kPa pressure at various ambient temperatures

From these results we can conclude that in case of PiCNTs(x)CB(y) piezoresistive sensitivity dependance of temperature diverse patterns were observed. This can be explained with more complex electrical grid structure and different CNTs and CB mobility during deformation, therefore temperature influence is determined by the dominant filler in the conductive structure.

In addition to these results PNCC electrical resistivity dependance of temperature is evaluated in range from – 100 °C to 60 °C. As can be seen from Fig. 6. PiTEG13 composition shows negative temperature coefficient (NTK) of electrical resistivity in range from -100 °C to -15 °C, however in range from 0 °C to 60 °C positive temperature coefficient (PTC) is observed. It is important to mention that at – 70 °C composite matrix transition from glassy state to hyperelastic state occurs and significant changes of linear thermal expansion coefficient is observed from $54{,}8 \cdot 10^{-6}$ K^{-1} to $325{,}1 \cdot 10^{-6}$ K^{-1} (determined by Ilze Aulika at University of Vienna, Institute of Experimental Physics).

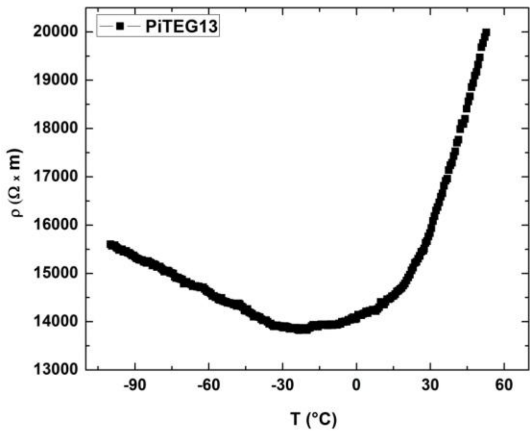


Fig. 6. PiTEG13 specific electrical resistivity dependance of temperature

In the literature NTK in polymer electroconductive filler composites is explained by electron hopping or tunneling trough potential barier. Mainly two mathematical models are used:

1) Sheng temperature fluctuation induced tunneling (TFIT) [2]

$$\rho = \rho_0 \exp\left(\frac{T_1}{T + T_0}\right) \quad (1.)$$

2) Motta variable range hopping of charge carriers (VHR) [3]

$$\sigma\sqrt{T} = \sigma_0 \exp\left(-\left(\frac{\mathrm{T}_0}{\mathrm{T}}\right)^{\frac{1}{4}}\right) \quad (2.)$$

PiTEG13 electrical resistivity dependance of temperature described with TFIT models and achieved values are shown in Fig.7. The same results where described with VRH model and achieved values are shown in Fig. 8.

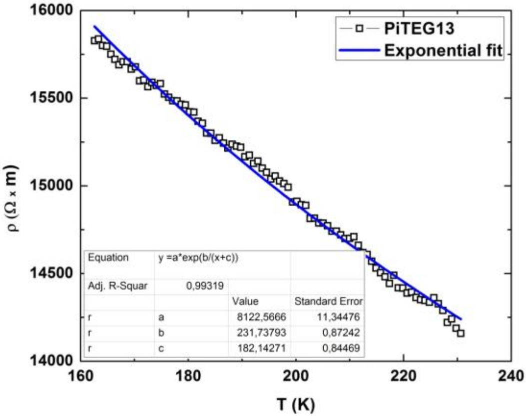


Fig. 7. PiTEG13 specific electrical resistivity dependance of temperature described with TFIT model

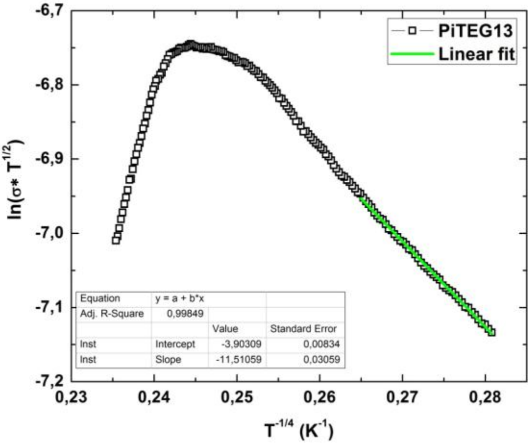


Fig. 8. PiTEG13 specific electrical resistivity dependance of temperature described with VRH model

In the doctoral work it was concluded that VRH model describes NTC of PiTEG and PiCNTs(x)CB(y) composites more precise and it is possible to achieve physically real values of fittings compared to TFIT model. However in the literature there are no models that describes PTK at higher temperatures. Therefore a new mathematical model is proposed that is based on differences between matrix and filler linear thermal expansion coeficients. PTK is described as tunneling current reduction due to increase of particle mutual distance increase with increase of temperature:

$$\ln R = lnR_0 + A_0\alpha\Delta T \quad (3.)$$

At larger particle separation conductive grid breakdown is observed and resisitivity increase is described as:

$$\ln R = lnR_0 + A\alpha\Delta T + B(\alpha\Delta T)^2 + C(\alpha\Delta T)^3 + D(\alpha\Delta T)^4 \quad (4.)$$

With these equations PiTEG and PiCNTs(x)CB(y) PTK are described. PiTEG13 composition results are shown in Fig. 9. From doctoral work experiments were concluded that PiTEG electrical resistivity dependance of temperature can be described with both equations, however PiCNTs(x)CB(y) only with 3rd equation. This was explained with PiCNTs(x)CB(y) conductive filler geometry which can make more electrical contacts between two particles, therefore in temperatures up till 60 °C conductive grid breakdown was not observed.

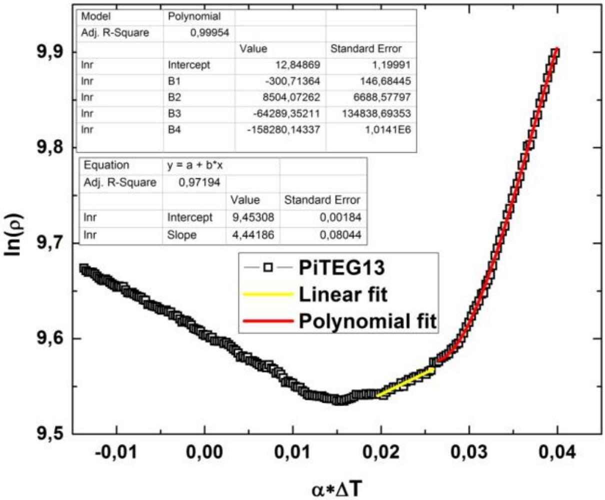


Fig. 9. PiTEG13 electrical resistivity dependance of temperature fitting with equations 3 and 4

The same electrical resistivity dependance of temperature fitting with various mathematical models was done for polycaprolactone/CNT composites (PCL/CNT). From Fig. 10, 11, and 12 one can see PCL/CNT fittings with good determination coefficients ($R^2 > 0,97$). These results and elaborated mathematical model are approbated in publication during dissertation [4].

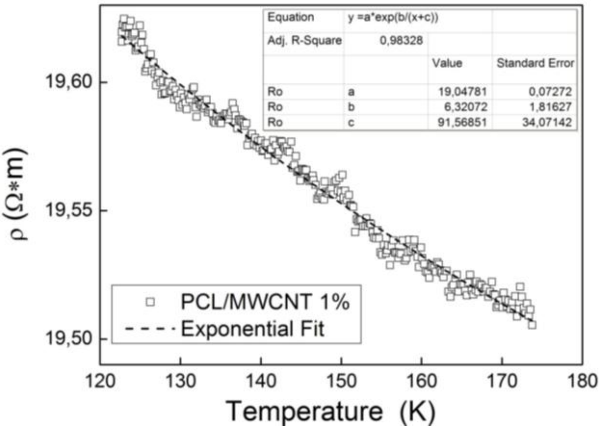


Fig. 10. PCL/CNT specific electrical resistivity dependance of temperature described with TFIT model

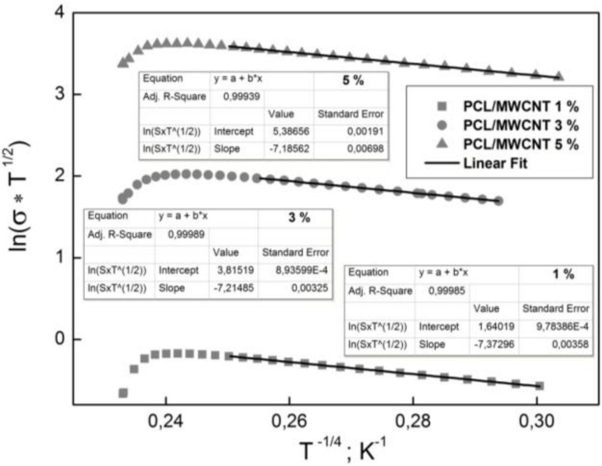


Fig. 11. PCL/CNT specific electrical resistivity dependance of temperature described with VRH model

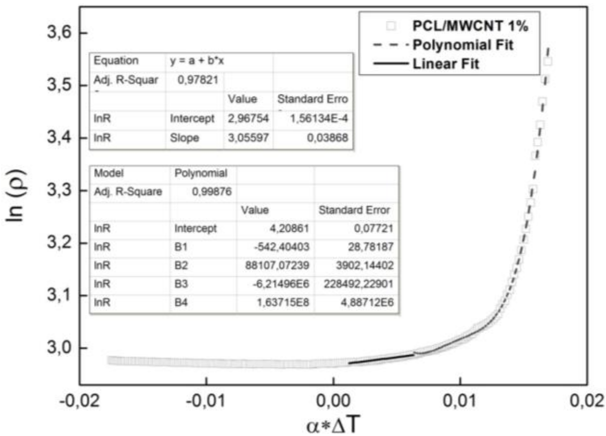
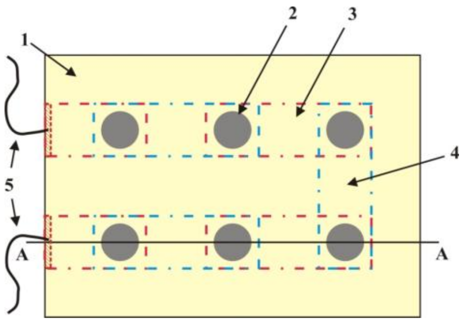


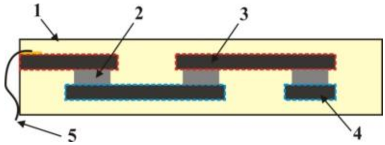
Fig. 12. PCL/CNT specific electrical resistivity dependance of temperature described with equation 3 and 4

At the 3rd subsection of experimental part 3 types of entirely hyperelastic sensor systems (VSSS) are elaborated where as a raw materials PNCC made in „Baltic Rubber Factory" (BGF) are used. These PNCC were used due to relatively large material amounts. 3 types of VSSS were made using layered composite design where pressure sensitive elements were jointed with electrode elements and incorporated into protective non-conductive natural rubber shell:

1) Sensor system that consist of 6 piezoresistive elements connected in series to provide better piezoresistive sensitivity (VPS) as shown in Fig. 13.



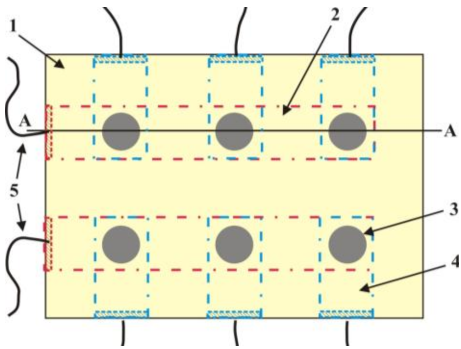
a) Planar schematic view of VPS



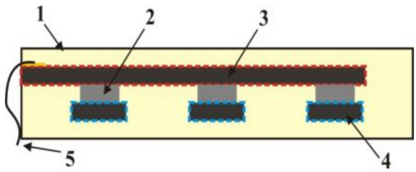
b) schematic view of AA cross section of VPS

Fig. 13. Planar schematic view of VPS and schematic AA cross section of VPS. consisting of : 1 – non-conductive outer shell, 2 – piezoresistive PNCC, 3 – upper layer of conductive PNCC, 4 – lower layer of conductive PNCC, 5 – wires

2) Sensor system that consist of 6 piezoresistive elements that could be monitured separately (APS) as shown in Fig. 14.



a) Planar schematic view of APS



b) schematic view of AA cross section of VPS

Fig. 14. Planar schematic view of APS and schematic AA cross section of APS. consisting of : 1 – non-conductive outer shell, 2 – piezoresistive PNCC, 3 – upper layer of conductive PNCC, 4 – lower layer of conductive PNCC, 5 – wires

3) VSSS with one piezoresistive element (PS) as shown in Fig. 15.

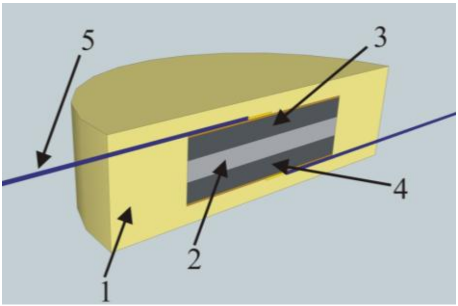
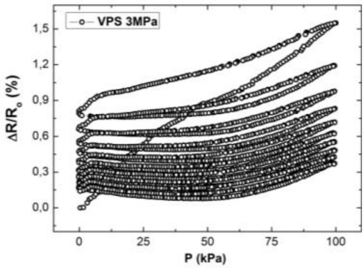
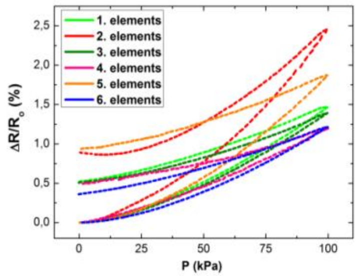


Fig. 15. Schematic view of PS cross section. consisting of : 1 – non-conductive outer shell, 2 – piezoresistive PNCC, 3 – upper layer of conductive PNCC, 4 – lower layer of conductive PNCC, 5 – wires

The piezoresistive effects of all VSSS in room temperatures are shown in Fig. 16. Temperature influence of PS sensitivity are also shown in Fig 16. Based from these results it was concluded that PS working temperature is from 20 °C to 85 °C, however for better response sensor temperature calibration must be used.



1



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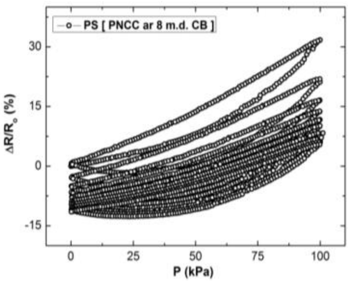

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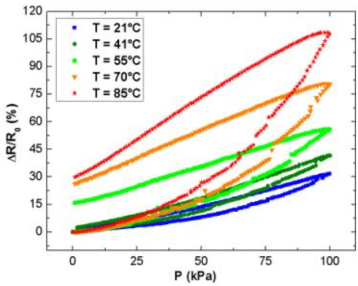

4

16. att. Piezoresistive sensitivity of VSSS under single exposure of 100 kPa pressure, where: 1 – VPS; 2 – APS; 3 – PS , 4 – PS sensitivity at various temperatures

Similarly as ambient temperature range evaluation, mechanical loading frequency play important part for VSSS applications. Unfortunatelly this dependance could not be determinied due to technical limitations. However the loading frequency influence on piezoresistive properties was determined for PNCC used as VSSS sensitive element in tensile mode at loading frequencies 0,005; 0,01; 0,05; 0,1; 3; 5; 10; 15; 20; 25; 30; 35; 40 and 45 Hz from 0 to 6,6% normal strain. Results are shown in Fig. 17 where one can see decrease of piezoresistive sensitivity with increase of loading frequency. This is explained due to conductive network structure reorganization. Respectively composite electrical structure changes due to deformation induced conductive filler mobility which in turn increase overall resistivity because the average distance between fillers increases and reduction of number of the electrical channels.However when the deformation is removed the electrical structure returns to the previous state. At higher mechanical loading frequencies return to the previous state in hindered and electrical resistivity at zero strain increases and overall structure consists of less channels. This reduction of channels leads to raise of overall resistivity at deformed state, but since the structure consist of less channels the piezoresistive effect decreases.

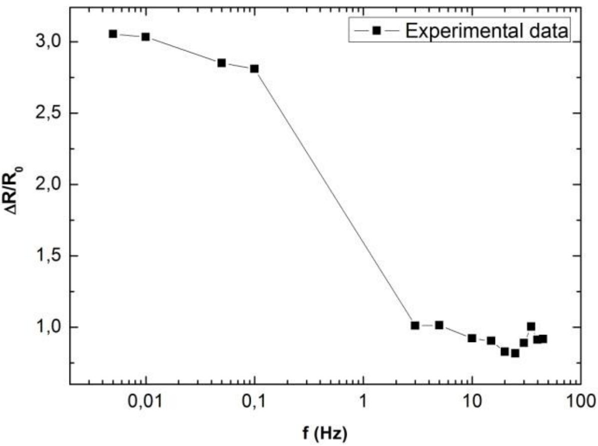

Fig. 17. VSSS sensorelement piezoresistive sensitivity dependance of loading frequency from 0 to 6,6 % normal strain

If all VSSS results are compared its possible to conclude that these mechanical sensors does not show direct dependence between mechanical input and resistivity output therefore precise values can not be determined. Although VSSS can be used in applications where it is necessary to detect or realatively evaluate the mechanical influence. Compared to other force or pressure sensors VSSS is hyperelastic and therefore is capable to detect forces in a very wide range from a gentle touch up to impact with a hard object. Evaluating advantages and disadvantages of VSSS we offer these VSSS applications:

1)Artificial skin or tactile sensors which would allow motorized equipments to sense interaction with the environment;

2)In security systems where it would be necesarry to determine the place where did mechanical influence occourred

3)For traffic or pedestrian flow monitoring to control the logistics more effective

4)In manufacturing plants as a smart switch, which would hold processes in case of accident to minimize total damage.

In the last subsection of experimental part device prototype for pressure detection with PS is elaborated with help from Einars Deksnis RTU, Robotics club. The device prototype (Fig. 18) is capable to moniture electrical resistivity of PS in time show it in graphical interface in 20 second time scale as well as count mechanical influences with variable parametrs.

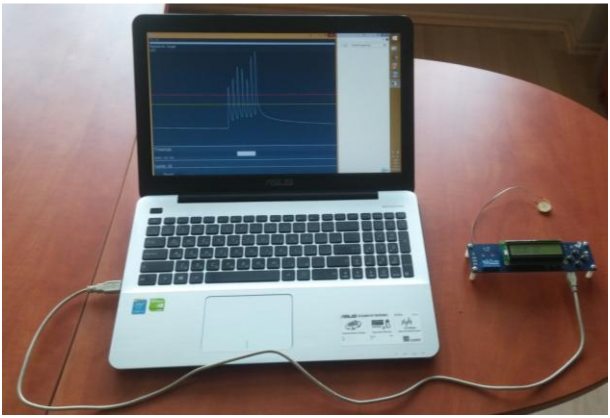

Fig. 18. Device prototype with PS and graphical interface

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[4] S.J.Chin, S.Vempati, P.Dawson, M.Knite, A.Linarts, K.Ozols, T.McNally. Electrical conduction and rheological behaviour of composites of poly(ε-caprolactone) and MWCNTs. *Polymer*, **2015,** 58, 209-221.

3. CONCLUSIONS

1) Polyisoprene / thermally exfoliated graphite composite has the highest piezoresistive sensitivity due to conductive filler geometry and possible mutual contacts between particles. Respectively between particles with high probability only one electrical contact can happen which contact surface area is reduced due to crossdeformation induced particle mobility as well as with reduced probability of new conductive channel formation.
2) Increase of ultrasonic treatment of thermally exfoliated graphite lead to reduction of filler size by two orders. Piezoresistive effect of composites made from these fillers showed better sensor response to uniaxial pressure due to better conductive filler mobility during deformation.
3) PiCNTs(x)CB(y) composites with specific filler concentrations showed better piezoresistive sensitivity compared to PiCB and PiCNTs. This is explained due to synergistic effect at specific both filler concentrations. CB as a 0 D nanomaterial exhibits better mobility in composite microstructure compared to 1D CNT leading to more complete conductive grid breakdown.
4) PiTEG showed increased piezoresistive sensitivity with increase of ambient temperatures due to reduction of polyisoprene tangent modulus. Therefore at the same mechanical stress larger deformations are achieved leading to reduction of tunneling currents.
5) Diverse patterns were observed of PiCNTs(x)CB(y) piezoresistive sensitivity dependance of ambient temperatures. This can be explained with more complex electrical grid structure and different CNTs and CB

mobility during deformation, therefore temperature influence is determined by the dominant filler in the conductive structure

6) The case of PiCNTs(4)CB(5) reduction of piezoresistive sensitivity with increase of temperature is due to CB greater impact of conductive structure / channels formation then CNTs since CB concentration and influence is proportianally higher then CNTs. Therefore at higher temperatures deformation induced CB conductive structure breakdown due to CB particle better mobility leads to new a conductive structure formation because CNTs are involved to the grid.
7) Electrical resistivity dependance of temperature has been determined for composites in range from -100 °C to 60 °C. Negative temperature coefficient in low temperatures has been quantitatively discribed with two mathematical models from literature. Positive temperature coefficient at higher temperatures is described with elaborated mathematical metod that is based on tunneling current reduction and subsequent brakedown of conductive channels due to increased mutual particle distances.
8) Three different entirely hyperelastic pressure sensor systems are elaborated. The active elements are manufactured from PNCC with various CB concentrations made in „BGF".
9) Cyclic mechanical frequency influence on piezoresistive sensitivity has been determined for composition which is used as sensitive element in sensor systems. Reduction of sensitivity with increase of frequency is due to hysteresis of filler mobility which leads to reduction of active channels that participates in piezoresistive effect.

APPROBATION IN PUBLICATIONS

Publications in journals or conference proceedings:

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