# PROSPECTIVE ELASTO-PLASTIC PRESSURE SENSORS
## *All-Elasto-Plastic Polyisoprene/Nanostructured Carbon Pressure Sensing Element*

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Abstract: Our further achievements in the design, processing and studies of physical properties of elastomer – nano-structured carbon composites as prospective compressive strain sensor materials for robotic tactile elements as well as for other automatic systems are presented. Composites made of polyisoprene matrix and high-structure carbon nanoparticle filler have been designed and manufactured to develop polymer nano-composites for flexible, entirely polymeric pressure sensing elements. Electrical resistance of the composites as a function of mechanical strain and pressure is studied. SEM pictures of cross-section surface of sensing elements are analyzed.

## 1 INTRODUCTION

In our previous study we have already shown the possibility to manufacture entirely flexible PNC sensing element with glued conductive rubber electrodes (Knite, 2008). Such elements show good mechano-electrical properties but they have one drawback – possible delamination of flexible electrodes during operation. In this study we further developed the technology of preparing all-elasto-plastic (AEP) strain sensing element with vulcanized flexible electrodes made of polyisoprene-nanostructured carbon (PNC) composite. Recently, some promising results have been presented regarding the application of polymer/conductive filler composites as strain and pressure sensors as well as selective gas sensors (Knite, 2002; Knite, 2004; Qu, 2007; Li, 2008; Knite, 2007; Sakale, 2009). Interesting and excellent properties have been obtained in case the composite contains dispersed nano-size conducting particles. If the size of carbon particle and specific surface area of carbon black are between 60 to 200 nm and 16-24 $m^2/g$, respectively (low-structure carbon nano-particles (LSNP)), the electrical resistance of natural rubber composites slowly decreases with applied pressure (Job, 2003). The effect is explained by the increasing number of conductive channels due to the increase of external pressure. Resistance of natural polyisoprene-carbon nanocomposites grows very rapidly and reversibly for both – tensile and compressive strain when high-structure carbon nano-particles (HSNP) (specific surface area 950 $m^2/g$, mean diameter 25 nm) are used as the filler (Knite, 2007). The sensing elements described in all mentioned papers contain metallic electrodes that reduce the flexibility of the whole element as well as delamination of electrodes can be possible due to bending. In this paper our recent success in the design, processing and studies of properties of vulcanized foliated composite sensor element is reported.

## 2 PREPARATION OF SAMPLES AND THE EXPERIMENT

The polyisoprene – nano-structured carbon black composite was made (see Figure 2) by rolling high-structure PRINTEX XE2 (DEGUSSA AG) nano-size carbon black (CB) and necessary additional ingredients (sulphur and zinc oxide) into a Thick Pale Crepe No9 Extra polyisoprene (MARDEC, Inc.) matrix and vulcanizing under 3 MPa pressure at 155 ˚C for 20 min. The mean particle size of PRINTEX XE2 is 30 nm, DBP absorption – 380 ml/100 g, and the BET surface area – 950 $m^2/g$.

The sensor element was made as follows. Two blends of polyisoprene accordingly with 30 and 10 phr (parts per hundred rubber) carbon black have been mixed. Initially 30 phr of PRINTEX have been



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used for obtaining PNC composite electrodes, but the tests of mechanical and electrical properties showed, that electrodes made from PNC composites with 20 phr of PRITEX were as much conductive as 30 phr carbon black/polyisoprene electrodes but had better elasticity as well as superior adhesion to active element. Three semi-finished rounded sheets made from mentioned above two PNC composite blends have been formed and fitted onto special steel die. Those are two sheets for conductive electrodes (30 phr CB) and one sensitive sheet (10 phr CB) for pressure-sensing part. Each of these three sheets were separately pre-vulcanized under 3 MPa pressure and 110°C temperature to obtain flat surfaces. This operation lasts 10 minutes. After that the components were cooled and cleaned with ethanol. Further, all three parts were joined together in one sensor element and were placed into the steel die and vulcanized under pressure of 30 MPa and 155° C temperature for 20 minutes vulcanization (previous attempts (Knite, 2008) to create sensor element with conductive glue were shown to be relatively ineffective). To study mechano-electrical properties small brass foil electrodes were added before vulcanization. Finally, disc shape sensor 50 mm in diameter and 3 mm thick was obtained. From this preparation we cut out useful sensor elements for testing (Figure 1).

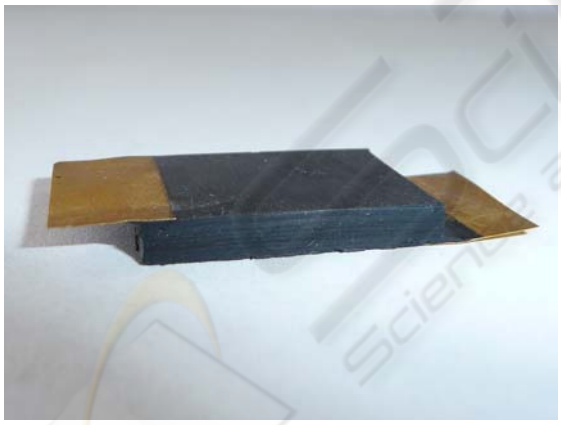

Figure 1: The accomplished all-elasto-plastic sensor element with brass foil extensions.

A modified Zwick/Roell Z2.5 universal testing machine, HQ stabilized power supply and a KEITHLEY Model 6487 Picoammeter/Voltage Source was used for testing mechano-electrical properties of sensor elements. All devices were synchronized with the HBM Spider 8 data acquisition logger. Resistance R versus compressive force $F$ was examined. Uniaxial pressure was calculated respectively.

# 3 RESULTS AND DISCUSSION

Before testing the accomplished sensor element, we measured the electrical properties of separate vulcanized electrode layers. We also separately tested the mechano-electrical properties of vulcanized active element layer to see whether it has expected sensing capabilities. The active element of the sensor (nano-structured carbon black composite with 10 phr) belongs to the region of the percolation threshold (specific electrical resistance $\rho = 12\ \Omega \cdot m$). The specific resistance for flexible electrodes is in the order of $0.1\ \Omega \cdot m$, which is noticeably above the percolation threshold.

Let's look closer at the conductivity properties of sensors. Measurement results for electrical resistance versus pressure for small pressure range are given in Figure 2.

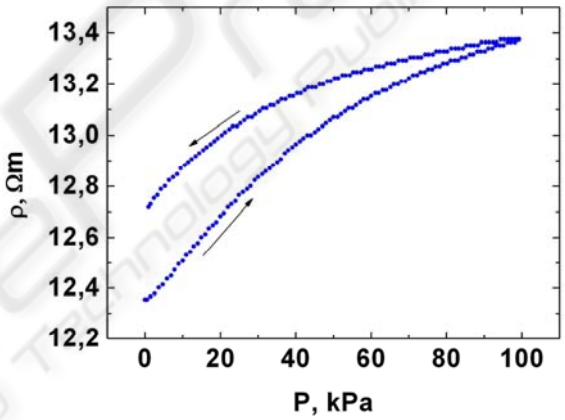


Figure 2: Electrical resistance of the all-elasto-plastic sensor element as function of pressure (lower pressure range, T = 294 $^0$K).

Measurement results for relatively large pressure range are shown in Figure 3. The observed positive piezoresistance effect can be explained by transverse slip of nano-particles caused by external pressure leading to disarrangement of the conductive channels. The volume concentration of conductor particles $V_C$ at which the transition proceeds is called the percolation threshold or the critical point. According to the statistical model, conductor particles, in the vicinity of $V_C$, assemble in clusters. Upon approaching $V_C$, the correlation radius $\xi$ (the average distance between two opposite particles of a cluster) diverges as

$$\xi \sim /V\text{-}V_C/^{-\nu} \qquad (1)$$

where ν is the critical index (Roldughin, 2000).

In the vicinity of the percolation threshold, electrical conductivity of the composite changes as:

$$\sigma \sim /V\text{-}V_C/^t \qquad (2)$$

where $t$ is the critical index (Roldughin, 2000). Under mechanical deformation of composites $\xi$ and, consequently, $\sigma$ change. This is the reason causing the piezoresistive effect.

Because of higher mobility of HSNP compared to LSNP the electro-conductive network in the elastomer matrix is easily disarranged by very small tensile, compressive or shear strain. We suppose this feature makes the elastomer–HSNP composite an option for flexible sensitive tactile elements for robots and automatics.

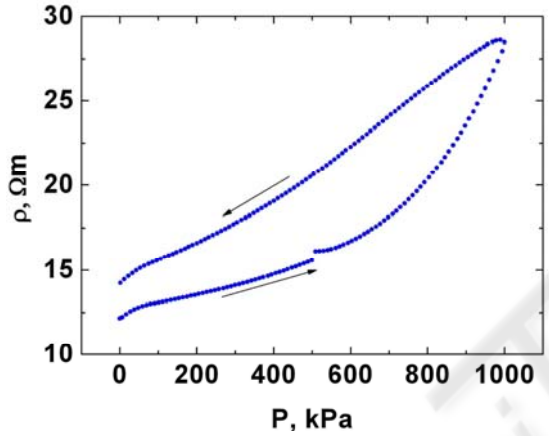


Figure 3: Electrical resistance of the all-elasto-plastic sensor element as function of pressure (higher pressure range, T = 294 $^0$K).

The scanning electron microscopy (SEM) was used to check the quality of joined regions of three PNC sheets of the AEP sensor element. SEM micrographs of breaking surface of the sensor element are shown in Figure 4. To prepare the sample for SEM investigations the sensor element was frozen in liquid nitrogen and then broken. Good quality of joining of all three PNC sheets can be clearly visible in SEM images with different scale (Figure 4). Pale regions correspond to electrically more conductive PNC composite with 30 phr CB and dark regions cover the PNC composite with 10 phr CB. The pale particles, which are visible in the bottom picture (Figure 4), are carbon nano-particles.

A functioning model of low-pressure-sensitive indicator was made. The block diagram of pressure indication circuit is shown on Figure 5. The sensor is connected to power supply (PS) via resistor (R) and to the input of amplifier (Amp). Transistor-based two-stage amplifier includes integrating elements.

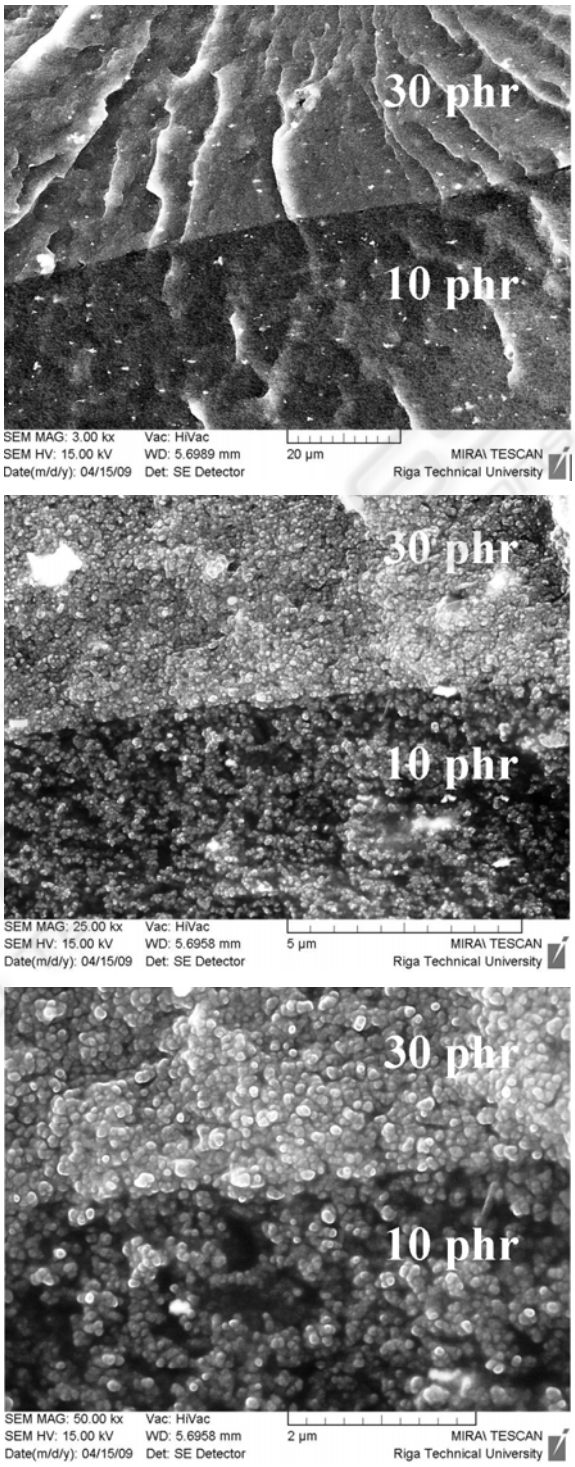


Figure 4: SEM micrographs of sensor element. Sensor element was frozen in liquid nitrogen and then broken in two. One of the broken sides is shown in different scales: 20 µm, 5 µm and 2 µm. Boundary between two PNC composite layers with 10 and 30 phr (parts per hundred rubber) carbon black are shown.

These elements are necessary to avoid noise from induced currents and to flatten the wavefronts. The first stage amplifies the signal in linear mode. The second stage works in saturation mode. The output of the amplifier is connected to the comparator (Comp), which forms sharp wavefronts.

These signals are passed to the differential circuit and they form a sharp pulse, which is passed further to the one-shot multivibrator (OSM).

The duration of the pulse of the OSM is adjustable. The OSM is necessary to form the determined length of pulse which is independent from AEP sensor element deformation time. The output of OSM is connected to performing device PD (indicator/counter or actuator).

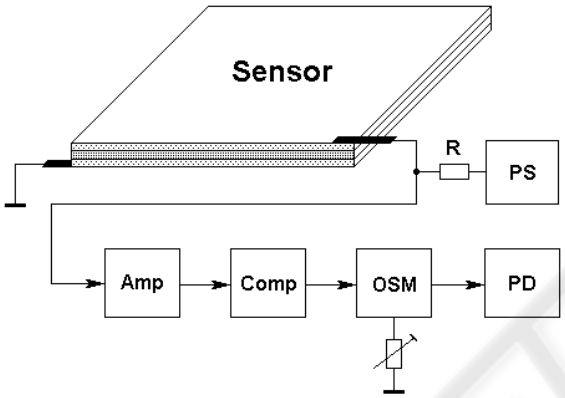


Figure 5: Block diagram of pressure-sensitive indication circuit with completely elasto-plastic sensing element.

## 4 CONCLUSIONS

Completely flexible polyisoprene – high-structured carbon black all-elasto-plastic sensing element has been designed, prepared and examined.

The sensor element was composed of two electrically conductive composite layers (electrodes) and piezoresistive PNC layer (active element) between them. A method for curing three-layer hybrid composite for pressure sensing application was developed. The joining in-between conductive flexible electrodes and sensitive sensor material was remarkably improved.

Hybrid three-layer polyisoprene/high-structure carbon black composite has shown good pressure sensing properties. Functioning model of low-pressure-sensitive indication circuit which can turn on suitable actuator has been made.

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