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Piezoresistive rubber nanocomposites for pressure sensing

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Summary

In this work we present piezoresistive polyisoprene - carbon nanocomposites (PICN) for pressure sensing applications. Latest results on designing, preparing and obtaining the PICN with necessary properties, as well as our efforts on practical application of such material are presented. The dependence of PICN sensing properties on vulcanization time, filler type and concentration is evaluated. The completely hyper-elastic multi layer structural design is proposed to be used as a prospective sensing element.

Introduction

Soft piezoresistive materials attract their attention because of their superior ability to be integrated into various non-solid structures and give wide response over various external parameters such as strain, pressure, bending and others. The key success of these materials is in fact, that they can be rather easily prepared using conventional rubber technology, only using specific ingredients and their quantitative formulation [1]. The specific properties of the final material can be tailored according to desired application.

The concept of research

To obtain piezoresistive PICN, the concentration of conductive filler must be within the limits of electrical percolation threshold [2,3]. To match this criteria, all the percolation thresholds for all types of PICN were determined at first. The critical percolation parameters were estimated using statistical percolation theory [4]. The evaluation of critical parameters gives indirect evidence about the type and main properties of conductive grid in PICN [5]. Then the basic conclusions can be made - if and how the type of filler and dispersing method used affects the final properties of PICN. The dependence of initial DC, AC and piezoresistive properties on vulcanization degree was also investigated for carbon black filled composites. The evaluation of these results together with IR spectra should give us a clear view how the vulcanization affects the electrical conductivity percolation. Finally, SEM images of PICN with different vulcanization times should give us an obvious evidence on the development of conductive grid and its properties.

Materials and methods

PICN were made by dispersing Degussa Printex Xe2 high structure carbon black or Multi Wall Carbon Nanotubes (MWCNT) into natural polyisoprene raw rubber. Various dispersing techniques were used, like roll mixing, chloroform solution mechanical mixing and chloroform dispersion mixing with ultrasound. The obtained raw rubbers were pressure vulcanized using hot steel mould. The samples with different vulcanization times were made. The sandpaper polished brass foil mould inserts were used to obtain reliable electrical contacts on samples. The initial DC electrical properties of samples were determined using simple digital multimeters, the high electrical resistivity was measured using Keithley Model 6487 picoammeter/voltage source, the piezorezistivity was determined using ZwickRoell Z2.5 universal material testing machine, coupled with Agilent A34970 digital data logger and the AC properties were measured using Agilent E4980A precision LCR meter. The Tescan/Mira LMU SEM was used for large magnification imaging. IR spectra were obtained on a Bruker Vertex 70 Fourier transform infrared spectrometer using attenuated total reflection (ATR) unit.

Conclusion

It was found, that the dimensional properties and aggregate structure of filler particles have noticeable affect on electrical and piezoresistive properties on PHSCBN. According to this, the appropriate dispersing method of filler should be chosen according to necessary final properties of the composite. The development of conductive grid in PHSCBN closely correlates with degree of vulcanization. The crosslinking plays major role on reversibility of piezoresistance as well as on initial electrical properties of PHSCBN.

The completely hyper-elastic multi layer structural design is proposed to be used as a soft pressure sensor.

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PIEZORESISTIVE RUBBER NANOCOMPOSITES FOR PRESSURE SENSING

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The INTRODUCTION Soft piezoresistive materials attract their attention because of their superior ability to be integrated into various non-solid structures and give wide response over various external parameters such as strain, pressure, bending and others. The key success of these materials is in fact, that they can be rather easily prepared using conventional rubber technology, only using specific ingredients and their quantitative formulation [1]. The specific properties of the final material can be tailored according to desired application.

In this work we present piezoresistive polyisoprene - carbon nanocomposites (PICN) for pressure sensing applications. Latest results on designing, preparing and obtaining the PICN with necessary properties, as well as our efforts on practical application of such material are presented. The dependence of PICN sensing properties on vulcanization time, filler type and concentration is evaluated. The completely hyper-elastic multi layer structural design is proposed to be used as a prospective sensing element.

The EXPERIMENTAL To obtain piezoresistive PICN, the concentration of conductive filler must be within the limits of electrical percolation threshold [2,3]. To match this criteria, the percolation thresholds for all types of PICN must be determined at first (Fig.1). The critical percolation parameters are estimated using statistical percolation theory (Fig.2-3) [4]. The evaluation of critical parameters gives indirect evidence about the type and main properties of conductive grid in PICN [5,6]. The dependence of initial DC, AC and piezoresistive properties on vulcanization degree was also investigated for carbon black filled PICN (Fig.4-6). The evaluation of these results together with IR spectra (Fig.7) should give us a clear view how the vulcanization affects the electrical percolation. Finally, SEM images of PICN with different vulcanization times should give us an obvious evidence on the development of conductive grid and its properties.

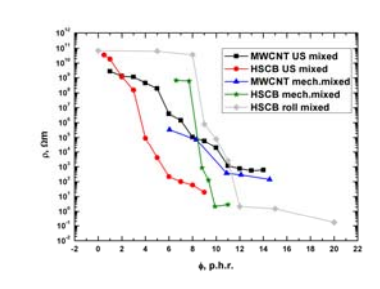

Fig.1 The electrical percolation thresholds of differently prepared PICNs with various fillers used.

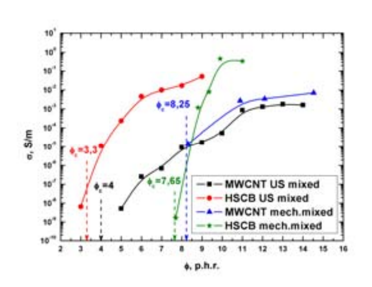

Fig.2 The critical electrical percolation concentrations (φ_c) of differently prepared PICNs with various fillers used.

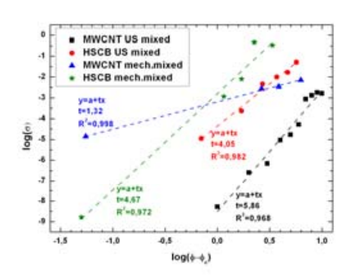

Fig.3 The estimated critical percolation indexes (t) of differently prepared PICNs with various fillers used.

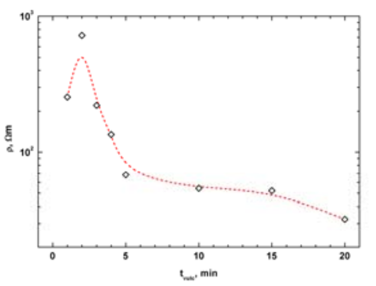

Fig.4 The dependence of initial electrical resistivity on vulcanization time for PICN filled with 10 p.h.r. of HSCB.

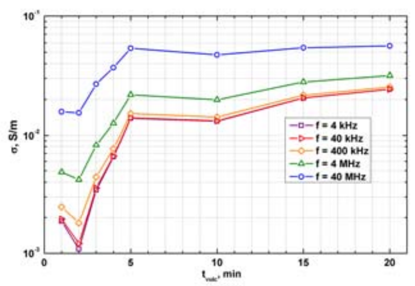

Fig.5 The dependence of AC conductance on vulcanization time for PICN filled with 10 p.h.r. of HSCB.

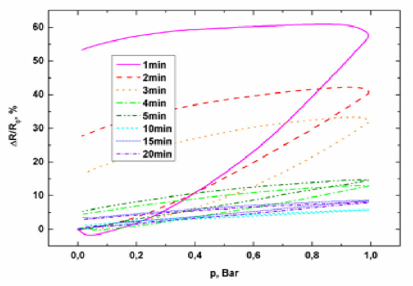

Fig.6 The dependence of the change of the resistivity on operational pressure for PICN filled with 10 p.h.r. of HSCB with gradually increased vulcanization times.

MATERIALS and METHODS PICN were made by dispersing Degussa Printex Xe2 high structure carbon black (HSCB) or Multi Wall Carbon Nanotubes (MWCNT) into natural polyisoprene raw rubber. Various dispersing techniques were used, like roll mixing, chloroform solution mechanical mixing and chloroform dispersion mixing with ultrasound. The obtained raw rubbers were pressure vulcanized using hot steel mould. The roll mixed CB filled PICN samples with different vulcanization times were made. The sandpaper polished brass foil mould inserts were used to obtain reliable electrical contacts on samples. The initial DC electrical properties of samples were determined using simple digital multimeters, the high electrical resistivity was measured using Keithley Model 6487 picoammeter/voltage source, the piezorezistivity was determined using ZwickRoell Z2.5 universal material testing machine, coupled with Agilent A34970 digital data logger and the AC properties were measured using Agilent E4980A precision LCR meter. The Tescan/Mira LMU SEM was used for large magnification imaging. IR spectra were obtained on a Bruker Vertex 70 Fourier transform infrared spectrometer using attenuated total reflection (ATR) unit.

DISCUSSION By performing determination of percolation thresholds, it was found out, that the dimensional properties and aggregate structure of filler particles have noticeable affect on electrical percolation properties of PICN. The sharpest percolation transition was obtained with roll mixed HSCB filled PICN **(Fig.1)**. The dispersing with ultrasound (US) can be used to noticeably lower the desired percolation threshold **(Fig.2)**. According to Jakubowicz [6], the critical index can be interpreted as an average number of electrical contacts, each conductive particle has with its neighbors. Thus US dispersed MWCNT PICN appears to have the most complex conductive grid and mechanically dispersed MWCNT the most simple one **(Fig.3)**. The structure of HSCB conductive grid appears to be relatively high for all HSCB filled PICN. The development of conductive grid in HSCB filled PICN closely correlates with its degree of vulcanization, which is evidenced by all - DC, AC and piezoresistance measurements **(Fig.4-6)**. As it is seen in **Fig.6**, the cross linking plays major role on reversibility of piezoresistance of HSCB filled PICN. The IR spectra of gradually cured HSCB filled PICN shows gradual decrease of C=C double bond peaks at 1667 cm-1 and 1539 cm-1 and subsequential increase of S-C bond peaks at 1076 cm-1 and 1135 cm-1 and S=C bond peaks at 1035 cm-1 with the increasing time of the vulcanization **(Fig.7)**. The SEM pictures of liquid nitrogen broken PICN shows gradual agglomeration of HSCB particles with increased vulcanization time **(Fig.8)**. Finally, the completely hyper-elastic multi layer structural design is elaborated and proposed to be used as a completely soft pressure sensor **(Fig.9)**.

Conclusions

→ It was found, that the dimensional properties and aggregate structure of filler particles have noticeable affect on electrical and piezoresistive properties on PICN.

→ The determination of critical percolation indexes for various PICN can be successfully used for indirect investigation of the complexity of the conductive grid.

→ The development of conductive grid in PICN closely correlates with degree of vulcanization.

→ The crosslinking plays major role on reversibility of piezoresistance as well as on initial electrical properties of PICN.

→ The completely hyper-elastic soft pressure sensor can be made from PICN.

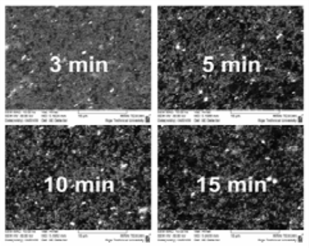

Fig.8 The SEM images of HSCB filled PICN under different times of vulcanization

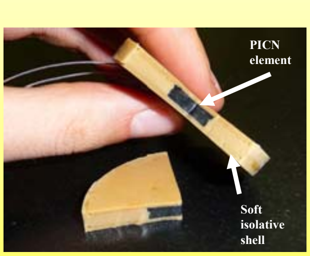

Fig.9 The radially cross cutted functional multilayer completely soft pressure sensing element prototype.

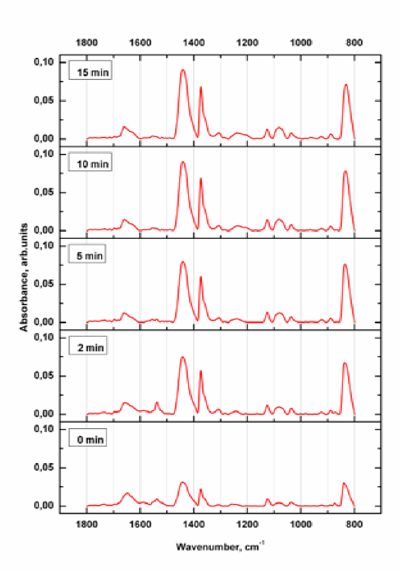

Fig.7 The FTIR spectra of HSCB filled PICN under gradually increased vulcanization time.

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