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2018

TUBALL^{TM} MATRIX 605 influence on HCR mechanical properties in the anti-static and conductive compounds

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Novosibirsk 2018.

PURPOSE OF THIS STUDY

Evaluation of TUBALL™ MATRIX 605 influence on mechanical properties of HCR(60 Shore A) based compounds. Two concentrations of TUBALL™ MATRIX 605 were tested to evaluate an impact to HCR in anti-static and conductive compounds accordingly – 0.8% (10^5-10^7 Ohm*cm) and 5% (below 10^2 Ohm*cm).

TESTING AND EQUIPMENT

Testing

Electrical properties:
ASTM D257 - Standard Test Methods for DC Resistance or Conductance of Insulating Materials
ASTM D991 – Rubber Property—Volume Resistivity Of Electrically Conductive and Antistatic Products.
Mechanical properties:
ASTM D412 – Properties in tension for Vulcanized rubber and Termoplastic Elastomers.
ASTM D624 – Standard Test Method for Tear Strength of Conventional Vulcanized Rubber and Thermoplastic Elastomers. Test method “B”.
ASTM D2240 – Standard Test Method for Rubber Property – Durometer Hardness, type A.
ASTM D7121 – Standard Test Method for Rubber Property—Resilience Using Schob Type Rebound Pendulum
ASTM D5963 - Standard Test Method for Rubber Property—Abrasion Resistance (Rotary Drum Abrader)
ASTM D5992 - Standard Guide for Dynamic Testing of Vulcanized Rubber and Rubber-Like Materials Using Vibratory Methods.

Equipment

- Two roll rubber mill LM 200/400, roll diameter – 200mm, length – 400mm.
- For curing rubber samples preparation curing press 40-250 1E was used.
- Post curing – laboratory oven SNOL-3,5.3,5.3,5/3,5-i5m was used
- Measurement of electrical resistivity according to ASTM D991 of vulcanized samples were carried out using the devices: the power supply "DC POWER SUPPLY HY 5003D", voltmeter "MNIPI B7-72", Pico ampere "MNIPI A2-4"
- Measurement of electrical resistivity according to ASTM D257 of vulcanized samples were carried out on teraommeter: TO-3 cable
- Tensile and tear properties – Shimadzu AGS-5kNXD.
- Hardness – Zwick 3130 digital hardness tester to Shore A.
- Resilience – Rebound resilience tester Zwick 5109
- Abrasion resistance – Rotating Drum (DIN) Abrasion Tester
- DMA 242 E NETZSCH.

Materials

TUBALL^{TM} MATRIX 605 lot# 17sil02N1.250;

High consistency silicone rubber (HCR) – Elastosil R401/60S;

Curing agent –Luperox F40P (contains 40% of 1,3(4)-bis(tert-butylperoxyisopropyl)benzene) in HCR Elastosil 401/60S.

Total weight of each compounded batch - 300g.

EXPERIMENTAL PART

Pre-mixed TUBALL^{TM} MATRIX 605 used for concentration 0.8 wt.% in HCR

Not pre-mixed (direct mixing) of TUBALL^{TM} MATRIX 605 used for concentration 5 wt.% in HCR.

Adding of modifiers

TUBALL MATRIX pre-mixing

Recipe for pre-mixing of TUBALL^{TM} MATRIX 605 at Table 1 is shown.

Table 1. Formulation of Pre-mix of TUBALL^{TM} MATRIX 605

Recipe	concentration, wt. %
HCR	90
TUBALL^{TM} MATRIX 605	10

First 10% of TUBALL^{TM} MATRIX 605 was diluted in HCR according to following procedure: Make the gap between rolls 3 mm. Pass compound throw two roll mill, form it into a tube, turn the sample on 90° and fed material between the rollers again - 30 passes.

Preparation of the compounds

Recipes of experimental samples at Table 2 are given. Both pre-mixed and not pre-mixed materials was mixed identically.

Table 2. Recipe of test compounds

Ingredient	reference	0.8 wt. % MATRIX	5 wt. % MATRIX
HCR, wt. %	95.5	87.5	90.5
Pre-mixed TUBALL^{TM} MATRIX 605 , wt. %	–	8	–
TUBALL^{TM} MATRIX 605 , wt. %	–	–	5
40 % concentrate of peroxide, wt. %	4.5	4.5	4.5
Total, wt. %	100	100	100

First stage

Luperox F40P concentrate first were dilluted in HCR on two roll rubber mill according to the following procedure: Make the gap between rolls 3 mm. Pass compound throw two roll mill, form it into a tube, turn the sample on 90° and fed material between the rollers again - 30 passes.

Second stage

Pre-mixed TUBALL^{TM} MATRIX 605 or pure TUBALL^{TM} MATRIX 605 (depending on the recipe) add to the compound from the first mixing stage according to the following procedure:
Make the gap between rolls 3 mm. Pass compound throw two roll mill, form it into a tube, turn the sample on 90° and fed material between the rollers again - 30 passes.

Samples preparation

The following cuing parameters was used: temperature – 160 ºC, time – 6 min, pressure – 200 kgf/cm^2. Ready sample is a plate with 145x145x2 mm size.
The following post-cuing parameters was used: temperature 200 ºC, time – 4 hours.

RESULTS

Electrical resistivity

Impact of abrasion treatment

Electrical resistivity test results before and after abrasion surface treatment at Table 3 are shown. Sample was erased by sandpaper P80 10 times in two ways (functional test).

Table 3 – Electrical resistivity before and after abrasion

	Before abrasion		After abrasion, Sandpaper P80			
	Without conductive paste		Without conductive paste		With conductive paste	
TUBALL™ MATRIX content, wt. %	Volume resistivity, Ohm·cm	Surface resistivty, Ohm/sq	Volume resistivity, Ohm·cm	Surface resistivty, Ohm/sq	Volume resistivity, Ohm·cm	Surface resistivty, Ohm/sq
0.8	$4\cdot10^5\pm3\cdot10^5$	10^9	$8\cdot10^5\pm3\cdot10^5$	insulator	$5\cdot10^5\pm2\cdot10^5$	10^9
5	$4\cdot10^1\pm3\cdot10^1$	10^3	$6\cdot10^1\pm4\cdot10^1$	10^5	$3\cdot10^1\pm9\cdot10^0$	10^4

After the surface treatment (rigid surface) contact surface between electrode and sample decrease and as a result ensures high contact resistance and not accurate measurements. At the Table 3 electrical resistivity of abraded samples (measured according to ASTM D257 by 2-point method) has higher resistivity value. Conductive paste which increase contact surface allows to decrease contact resistance and perform more accurate measurements.

Impact of elongation

Electrical resistivity test results before and after elongation at Tables 4, 5 are shown. Sample was stretched on 50, 100, 300% and hold during different time. Stretching speed was 500 mm/min. After that, the stretching was removed and electrical resistivity was measured.

Table 4 – Electrical resistivity after stretching 0.8wt.% TUBALL™ MATRIX 605

Volume resistivity, Ohm·cm Initial, before stretching	Volume resistivity after stretching, Ohm·cm		
	50%	100%	300%
$4\cdot10^5$	0 Hours after stretching		
	$1\cdot10^8$	$2\cdot10^{11}$	$1\cdot10^{12}$
	24 hours after stretching		
	$7\cdot10^7$	$2\cdot10^{11}$	$6\cdot10^{11}$
	72 hours after stretching		
	$6\cdot10^7$	$1\cdot10^{11}$	$4\cdot10^{11}$
	96 hours after elongation		
	$7\cdot10^7$	$1\cdot10^{11}$	$5\cdot10^{11}$

Electrical resistivity of anti-static compounds increases on 3 orders of magnitude by stretching at 50 % and slightly decreases after relaxation of the sample.

Under higher deformations on 100 and 300% resistivity increases significantly on 6 orders of magnitude and not impacted after relaxation.

Table 5 – Electrical resistivity after stretching 5wt.% TUBALL™ MATRIX 605

Volume resistivity, Ohm·cm Initial, before stretching	Volume resistivity after stretching, Ohm·cm		
	50%	100%	300%
$4 \cdot 10^1$	0 Hours after stretching		
	$6 \cdot 10^1$	$8 \cdot 10^1$	$2 \cdot 10^2$
	24 hours after stretching		
	$5 \cdot 10^1$	$6 \cdot 10^1$	$1 \cdot 10^2$
	72 hours after stretching		
	$4 \cdot 10^1$	$6 \cdot 10^1$	$1 \cdot 10^2$
	96 hours after elongation		
	$4 \cdot 10^1$	$6 \cdot 10^1$	$1 \cdot 10^2$

Electrical resistivity of conductive compounds is not impacted by stretching at 50 and 100% and increase by one order of magnitude at the stretching at 300%. Resistivity value decreases after relaxation of the stretched samples.

Mechanical properties

Tensile characteristics

Tensile properties at Table 6 are shown.

Table 6 – Mechanical characteristics

MATRIX content, wt. %	M 50, MPa	M 100, MPa	M 200, MPa	M 300, MPa	Tensile Strength, MPa	Elongation at break, %
Reference	1.2±0.2	1.6±0.1	2.8±0.1	4.6±0.2	11.2±0.3	533±27
0.8	1.2±0.1	1.5±0.1	2.4±0.1	4.1±0.1	11.5±0.8	555±17
5	1.1±0.1	1.9±0.1	3.0±0.1	4.8±0.2	10.4±0.3	505±21

TUBALLTM MATRIX 605 has no significant impact on mechanical properties at the both levels of conductivity.

Tear characteristics

Tear properties at Table 7 are shown.

Table 7 – Tear strength

MATRIX content, wt. %	Tear strength, kN/m
Reference	19.9±0.6
0.8	21.9±1.1
5	21.2±1.2

TUBALL™ MATRIX 605 has no significant impact on tear characteristics at the both TUBALL™ MATRIX loading levels.

Abrasion resistance

Abrasion properties at Table 7 are shown.

Table 7. – Abrasion test results

MATRIX content, wt. %	Abrasion volume, mm^3	ARI, %
Reference	63.7±4.6	232±16
0.8	41.8±4.0	375±38
5	43.2±4.6	343±33

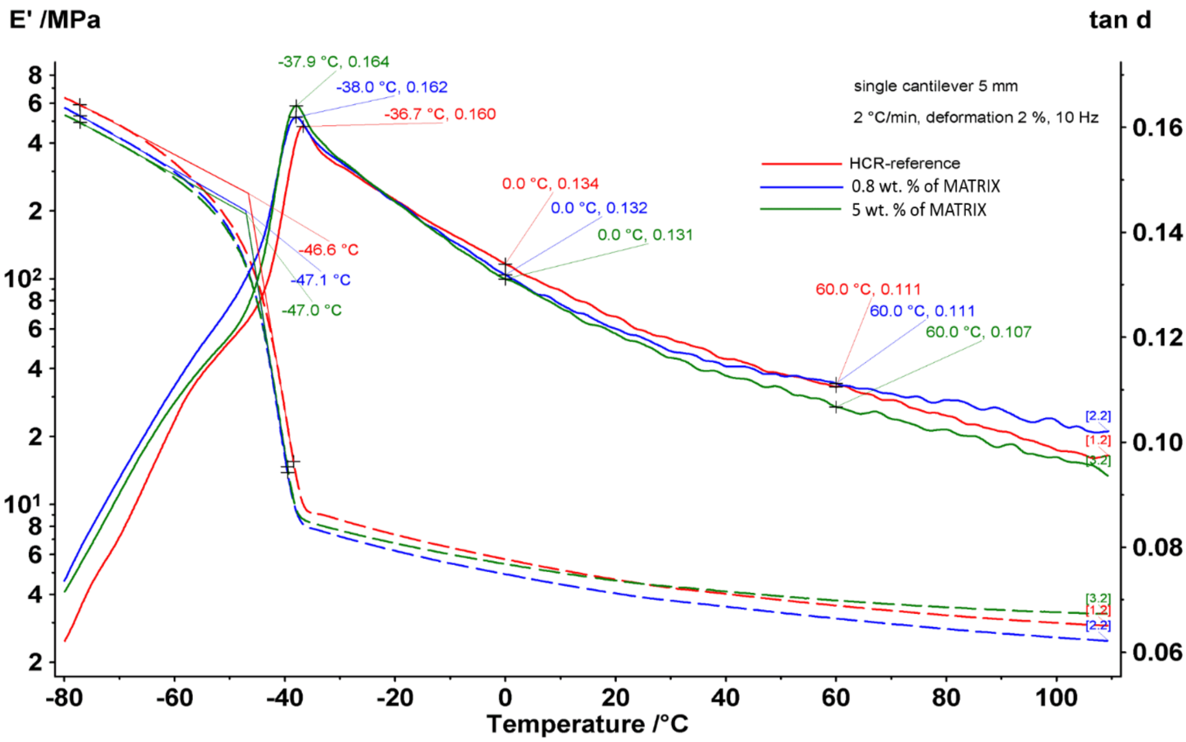
After TUBALL™ MATRIX 605 addition abrasion resistance of samples increased by 34%.

Dynamic mechanical analysis

Dynamic mechanical analysis results at figure 1 are shown. Testing conditions:

- single cantilever 5mm size;
- heating speed 2 ºC/min;
- deformation 2 %;
- frequency 10 Hz.

Figure 1. – DMA test results



TUBALL™ MATRIX 605 has no significant impact on dynamic properties.

CONCLUSIONS

TUBALL™ MATRIX 605 addition in concentrations 0.8% (10^5-10^7 Ohm*cm) and 5% (below 10^2 Ohm*cm):

- Allows to achieve wide range of electrical resistivity values depending on dosage;
- Electrical resistivity does not affected by abrasion treatment of surface. Conductive paste which increase contact surface on a rigid surface allows to decrease contact resistance and perform more accurate measurements;
- Electrical resistivity under dynamic deformation:

- For anti-static compounds increases on 3 orders of magnitude by stretching at 50 % and slightly decreases after relaxation of the sample. Under higher deformations on 100 and 300% resistivity increases significantly on 6 orders of magnitude and not impacted after relaxation.
- For conductive compounds is not impacted by stretching and decreases after relaxation of the samples.

Stability of conductive properties under dynamic deformations depends on loading of TUBALL™ MATRIX. Higher dosage of TUBALL™ provides stable conductive network and performance under deformations.

- No significant influence on such mechanical properties as tensile parameters, elastic properties, tear and dynamic characteristics;
- Abrasion resistance of samples increased by 34%.