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Antistatic polyurethane coats with hybrid carbon nanofillers

Summary – Thermoplastic coats based on commercial polyurethane aqueous dispersion and carbon nanofillers (graphite nanoplatelets and/or carbon nanotubes) were prepared and analyzed. Surface resistivity of the samples was in a range of 10^6 - $10^8 \Omega$ (<1 wt. % of carbon nanofillers). An incorporation of carbon nanofillers into coating systems improves hardness and thermal resistance of dry coats, whereas their glass transition temperature is not affected.

Keywords: waterborne, coating composition, varnish, coat, polyurethane, graphite nanoplatelets, carbon nanotubes

Antystatyczne powłoki poliuretanowe modyfikowane hybrydowymi nanonapełniaczami węglowymi

Streszczenie – Otrzymano i przebadano zestaw antystatycznych powłok bazujących na komercyjnej wodnej dyspersji poliuretanowej oraz nanonapełniaczach węglowych, tj. grafenie wielowarstwowym i/lub nanorurkach węglowych. Obecność nanonapełniaczy węglowych w ilości poniżej 1 % wag. nadaje powłokom rezystywność powierzchniową na poziomie 10^6 - $10^8 \Omega$, wyższą twardość i odporność termiczną, nie zmieniając znacząco temperatury zeszklenia matrycy poliuretanowej.

Słowa kluczowe: wodorozcieńczalny, kompozycja powłokotwórcza, lakier, powłoka, poliuretan, grafen wielowarstwowo, nanorurki węglowe

INTRODUCTION

The addition of carbon nanofillers enhances mechanical, electrical and thermal properties of polymer materials. Graphene (GN) exhibits high thermal and electrical conductivity,

high transparency and, moreover, its manufacturing is potentially less expensive than carbon nanotubes (CNT). Generally graphene is most often commercially prepared from graphite and offered in form of layered agglomerates [1]. Such carbon nanofillers are often called graphite nanoplatelets (GNP). Nevertheless, CNTs are still the most popular and useful carbon nanofiller applied in polymeric materials. These carbon structures, mainly multi-walled type, are widely offered by a number of commercial institutions in powdered form and as aqueous dispersions. Despite this there are very few announcements about polyurethane-based coating materials (PU) with carbon nanofillers, so a brief overview of the topic is presented below.

Although unfilled polyurethane coats usually exhibit good thermal, mechanical [2] as well as electrostatic properties (especially a in case of ionomeric PU) [3], an introduction of carbon nanoparticles significantly improves these features, and sometimes allows to keep a transparency on a high level of ca. 80 % [4-9]. Polyurethane compositions filled with CNT, GNP or Gn are described in literature, but they are generally applied as cast films. There are no reports on hybrid CNT/GNP carbon nanostructures application in polyurethane coating materials.

Ha and Kim investigated polyurethane-acrylate coats modified with multi-walled carbon nanotubes (MWCNTs) and crosslinked via UV irradiation [4]. Low content of MWCNT (0.1 wt. %) diminished electrical resistivity by about 7 orders. Moreover, presence of MWCNT decreases yield of UV photocuring reaction.

Song et al. prepared polyurethane coats with tolylenediisocyanate-functionalized MWCNTs [5]. An addition of 1 wt. % of nanofiller improved abrasion resistance by about 100 % as well as surprisingly increased the value of friction coefficient.

Jung et al. produced PU films using graphene formed from expandable graphite after long-lasting sonication process in N-methylpyrrolidone (NMP) [6]. PU solution in NMP and

GN/NMP dispersion were mixed together for 24 h and cast. The final films with 0.1 wt. % of GN (thickness 150 μm) exhibited high transparency (85 %) and electrical conductivity ($2 \cdot 10^3 \text{ S/cm}$).

Liao et al. obtained solvent-borne polyurethane-acrylate coats with graphene. Electrical percolation threshold was achieved for very low, i.e. 0.15 wt. % loading of GN [7].

Water-borne polyurethane coats with covalently functionalized GN were prepared using sol-gel method by Wang et al. [8]. Increment of tensile strength (70 – 90 %) and Young's modulus of polyurethane matrix with 2 wt. % content of Gn was observed.

Polyurethane two-component solvent-borne coating system, covalently bounded with silicone-modified graphene, was obtained by Ma and coworkers [9]. An incorporation of 0.2 wt. % of functionalized GN increased tensile strength (ca. 200 %), elongation at break (70 %), thermal degradation temperature (50 $^{\circ}\text{C}$), thermal conductivity (40 %) and reduced glass transition temperature (T_g) by 9 $^{\circ}\text{C}$ in comparison to the reference coat.

Yu et al. prepared UV-cured polyurethane-acrylate coats with covalently functionalized graphene oxide. An incorporation of mentioned carbon nanoparticles in amount of 1 wt. % increased thermal degradation temperature (17 $^{\circ}\text{C}$), tensile strength (73 %) and storage modulus of polymeric matrix [10].

The aim of this work was the preparation of water-borne coating compositions and coats based on commercial polyurethane aqueous dispersion and carbon nanofillers, i.e. carbon nanotubes as well as hybrid CNT/GNP systems, exhibiting electrical surface resistivity below $10^8 \Omega$, i.e. classifying such coats as antistatic materials.

EXPERIMENTAL

Materials

The following materials were used:

- Incorez W2205, commercial aqueous dispersion of polyurethane with polycarbonate backbone, 40 wt. % of solids, viscosity 125 mPa·s, contains N-ethylpyrrolidone and diethylene glycol monobutyl ether as cosolvents (Incorez, UK);
- Aquacyl AQ0101, 1 wt. % aqueous dispersion of multi-walled carbon nanotubes NC7000, average length 1.5 μm (Nanocyl, Belgium);
- ZUT-CNT, 1 wt. % aqueous dispersion of NC7000 carbon nanotubes, prepared at Polymer Institute (ZUT in Szczecin) via sonication process;
- ZUT-GNP, 0.7 wt. % aqueous dispersion of commercially available graphite nanoplatelets (650-700 m^2/g , 10-15 carbon layers, Cheap Tubes Inc., USA), prepared at Polymer Institute via sonication process;
- BYK-345, silicone surfactant for aqueous coating compositions (BYK-Chemie, Germany).

Sample preparation

Water-borne polyurethane varnishes with carbon nanofillers were prepared as follows. Polyurethane aqueous dispersion was mixed with BYK-345 in amount of 0.1 wt. part of surfactant / 100 wt. parts of coating composition by mechanical stirring (300 rpm, 15 min). Then, a proper amount of nanofiller dispersion (Aquacyl, ZUT/CNT and/or ZUT-GNP) was added and composition was mixed (450 rpm, 30 min). After that, coating composition was applied onto glass or poly(ethylene terephthalate) (PET) substrate using gap applicator (Zafil, Poland) and dried for 24 h at RT. Formulation scheme was graphically presented in Fig. 1. Four series of samples with different carbon nanostructures content (CNT or CNT/GNP) were prepared; the composition and sample symbols were presented in Table 1.

Fig.1; Table 1

Methods

Electrical surface resistivity of dry coats (on a glass substrate) was measured using Electrometer 6517A with electrode set Keithley 8009 (Keithley Instruments, Inc.) according to the Polish standard PN-E-04405:1998 (at 10 V). The pendulum hardness (three measurements of each sample) and the gloss value at 20 ° were tested on dry coats (glass substrate) using König pendulum (AWS-5, Dozafil, Poland) according to the Polish standard PN-EN ISO 1522:2008 and Micro-TRI-gloss μ (BYK-Gardner GmbH, Germany; ISO 2813), respectively. The digital images of surface micrograph of coat on glass substrate were prepared using Laser Scanning Microscope (LSM) (VK-9700, Keyence, USA). Transparency of cured coats (on a PET foil) was evaluated by using UV-Vis spectroscopy (Specord M40, Medson, Germany). Moreover, glass transition temperatures (T_g) of coats were evaluated by Differential Scanning Calorimetry (DSC) and Dynamic Mechanical Analysis (DMA) methods. DSC analysis was performed on small sample (ca. 10 mg) of dried nanocomposite coats; standard Al pans were used with scanning range $-20 \div 220$ °C and heating rate of 5 °C/min (DSC Q100, TA Instruments, USA). DMA tests were taken in the following conditions: tension test in a range of $0 \div 140$ °C, heating rate 3 °C/min, 1 Hz of frequency, amplitude 15 μ m (DMA Q800, TA Instruments). The T_g values of samples (PET substrate) on a basis of tan delta peak temperature were determined.

Thermogravimetric Analysis (TGA) was made using Q5000 thermoanalyzer (TA Instruments). Small doses (ca. 10 mg) of dried coats were examined in air atmosphere. Temperature range was $20 \div 800$ °C with heating rate of 5 °C/min.

RESULTS AND DISCUSSION

Electrical surface resistivity of coats

The surface resistivity values of polyurethane coats with CNT or CNT/GNP mixtures were shown in Table 2. Additionally, an influence of CNT content on surface resistivity of dry coats was graphically presented in Fig. 2. As can be seen, the addition of small amount of

CNT (1 wt. %) significantly decreased surface resistivity of polyurethane coats from $9 \cdot 10^{14} \Omega$ (reference sample RS-0) to $5.3 \cdot 10^7 \Omega$ (coat with Aquacyl dispersion; A-1.0) and $7 \cdot 10^6 \Omega$ (coat with ZUT-CNT dispersion; Z-1.0). Generally, the higher content of CNT, the better electrical conductivity. It should be noted that coats with ZUT-CNT dispersion exhibited distinctly better electrical properties at CNT concentration over 0.6 wt. % than samples with commercial aqueous dispersion of Nanocyl NC7000 (i.e. Aquacyl AQ0101). The established $10^8 \Omega$ threshold (recommended for antistatic materials) was achieved for samples with ca. 0.75 wt. % of ZUT-CNT (solid content) or 0.9 wt. % of Aquacyl, as presented in Fig. 2. Fig. 3 showed the CNT and GNP concentration influence on electrical resistivity of dry coats prepared using total 1 wt. % of either Aquacyl/ZUT-GNP or ZUT-CNT/ZUT-GNP mixtures. Although some differences for resistivity of coats containing $0.1 \div 0.5$ wt. % of CNT (and $0.9 \div 0.5$ wt.% of GNP) could be observed, samples with 0.6 wt. % of CNT and 0.4 wt. % of GNP reached ca. $10^8 \Omega$ for the analyzed parameter irrespective to the type of CNT dispersion (i.e. $4.1 \cdot 10^7 \Omega$ for A/GNP-0.6/0.4 and $7.3 \cdot 10^7 \Omega$ for Z/GNP-0.6/0.4). Nevertheless it should be described that coats with $0.6 \div 0.9$ wt. % of CNT (from Aquacyl) and $0.4 \div 0.1$ wt. % of GNP exhibited similar (or slightly reduced) electrical resistivity in relation to the A-1.0 coat filled with 1 wt. % of CNT (incorporated as a mentioned dispersion). In the case of coating systems based on ZUT-CNT dispersion of carbon nanotubes, the addition of GNP (even 0.1 wt. % instead of CNT, Fig. 3) increases that electrical parameter value. The presented results showed that CNT addition had greater (positive) influence on electrical properties of coats than GNP, however a small dose of GNP (in presence of CNT from Aquacyl dispersion) could maintain/improve the conductivity of polyurethane coats. The mentioned phenomenon was not analogically observed for dry coats prepared using ZUT-CNT and ZUT-GNP dispersions. Probably the CNT in the former dispersion were dissipated much better than in Aquacyl system, thus the

Table 2, Fig. 2, 3

addition of two-dimensional GNP to the coating composition based on ZUT-CNT did not significantly affect on electrical conductivity of dry polyurethane coat.

Other coats properties

The hardness test results were presented in Table 2. These values varied between 100 and 109 units. It was revealed that addition of carbon nanostructures improved to some extent the hardness of obtained coats. Interestingly, in the case of coats based on Aquacyl dispersion the best improvement of analyzed parameter was noted for sample with the highest CNT content (105 units; A-1.5), while this hardness value was registered for coats containing only 0.5 wt. % of ZUT-CNT dispersion. Moreover, taking into consideration the coating system filled with CNT/GNP hybrid nanofiller, generally higher hardness values were recorded for samples containing either higher amount of CNT from Aquacyl dispersion, (e.g. A/GNP-0.6/0.4) or lower amount of CNT from ZUT-CNT dispersion (Z/GNP-0.4/0.6 and Z/GNP-0.2/0.8 samples).

The LSM observations concerned samples with different GNP and/or CNT content.

Micrographs of prepared polyurethane coats were very similar; for example two images with various magnification of the Z/GNP-0.6/0.4 sample (0.6 wt. % of CNT and 0.4 wt. % of GNP) were presented in Fig. 4. Even though darker (CNT, GNP) and brighter areas (binder) were visible, the carbon nanofillers were quite uniformly dispersed in the polymeric matrix. It was confirmed by a relatively low electrical resistivity of all coats filled with the tested nanofillers.

Fig.4

The gloss assessment was performed on coats modified with either ZUT-CNT dispersion or hybrid CNT/GNP systems; the results were graphically presented in Fig. 5. As can be observed, the carbon nanofillers significantly reduced the coats gloss; in the case of coats with CNT the highest gloss value was registered for Z-0.5 (88 %) and the lowest for Z-1.5 (74 %). On the other hand, the GNP deteriorated the gloss parameter in a lower extent than

Fig.5

CNT, as it can be seen for samples with 1 wt. % of CNT (Z-1.0) or CNT/GNP mixture. All coats containing simultaneously both types of carbon nanofillers exhibited higher gloss values (79 ÷ 82 units) than mentioned sample filled with CNT, only (78 units). The highest gloss value among coats with 1 wt. % total content of carbon nanofillers was registered for Z/GNP-0.2/0.8 coat.

The CNT and CNT/GNP nanofillers addition reduced the dry coat transparency, as well. This parameter test results for selected samples, i.e. with electrical surface resistivity values below $10^8 \Omega$ were presented in Table 2. It can be seen that CNT presence in dry coat directly affected the analyzed feature (the higher CNT content, the lower transparency). It should be noted that samples based on Aquacyl and ZUT-GNP dispersions (i.e. coats with 1 wt. % of these nanofillers mixture) reached higher transparency (38 ÷ 39 % for A/GNP-type samples) than coat with 1 wt. % of the mentioned CNT (0 wt. % of GNP; A-1.0; 35 %). In the case of samples with either ZUT-CNT (Z-1.0; 37 %) or ZUT-CNT/ZUT-GNP mixture (38 ÷ 39 %) that phenomenon was not observed. The similar effect of Aquacyl and ZUT-CNT dispersions addition was registered for the surface resistivity results, i.e. GNP improved properties of Aquacyl-modified coats, but not the ZUT-CNT-based samples.

The glass transition temperatures were measured for samples based on ZUT-CNT and ZUT-GNP dispersions. The results determined by DSC and DMA techniques were compiled in Table 3. Moreover, detailed DSC thermographs of samples with CNT/GNP mixtures were presented in Fig. 6. As can be seen an incorporation of carbon nanofillers into the polyurethane matrix did not significantly affect the T_g of dry coats measured by DSC technique. Although the values of that parameter for RS-0 and modified coats were in a narrow range of 14.3 ÷ 15.7 °C (DSC technique), span of T_g values (DMA) in some cases was markedly higher (31.0 ÷ 36.5 °C). It means that tested carbon nanofillers did not

Table 3; Fig.6

significantly change the thermal features (analyzed using DSC), but only affected the thermomechanical properties of the polyurethane coats (measured by DMA). Differences between glass transition temperature values determined by these techniques are based on various nature of T_g determination. In DSC method the increase in specific heat of the material as it passes from the glossy to the rubber state is analyzed. DMA measures the viscoelastic properties of the material as a function of frequency and of oscillatory deformation. In the glassy state the material will exhibit solid-like characteristics and has a measurable shear modulus. The glass transition can be detected as the temperature where the storage modulus starts to fall rapidly (or tan delta reaches maximum value) with increasing temperature. Thus T_g transitions during DMA measurement is a kinetic rather than equilibrium phenomena. Differences between T_g values determined by DSC and DMA can vary as much as 15 °C (when based on storage modulus changes) up to 25 °C (tan delta peak evaluation) [11].

A similar set of samples was analyzed by TGA. The results of thermal stability of polyurethane coats with carbon nanofillers, i.e. temperatures at 5 % (T_5) and 10 % (T_{10}) mass losses were shown in Table 3. It was revealed that carbon nanostructures significantly improved the thermal stability of tested coats in temperature range of 100 ÷ 240 °C (Fig. 7). Moreover, the influence of GNP on observed phenomenon was more significant than of CNT. The T_{10} value for RS-0 was ca. 170 °C and rose up to ca. 208 °C (Z-1.0) and ca. 240 °C after 0.2 wt. % of CNT and 0.8 wt. % of GNP addition (Z/GNP-0.2/0.8). The growth of T_5 for mentioned samples was also observed: the increment was 12 °C (1 wt. % of CNT) and 85 °C for sample with the highest dose of GNP.

Fig.7

CONCLUSIONS

The water-borne varnishes and coats based on thermoplastic polyurethane aqueous dispersion and either CNT or CNT/GNP nanofillers mixtures were prepared and tested.

The content of nanoparticles up to 1 wt. % is responsible for electroconductive paths formation and decreasing surface resistivity of coats by 8 orders of magnitude in relation to the unfilled coat (i.e. to $10^6 \Omega$). It is important, that carbon nanostructures presence improves hardness and thermal stability of dry coats. Although the influence of GNP on electrical resistivity/conductivity is lower than of CNT, an addition of the former nanofiller (instead of CNT) cause increase of gloss and thermal stability of the analyzed materials. It is noteworthy that CNT and CNT/GNP-based coats preparation process is simple and possible to perform in a larger scale.

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TABLES

Table 1. Composition of polyurethane coats based on Incorez W2205 aqueous dispersion and carbon nanofillers

Tabela 1. Skład powłok poliuretanowych z wodnej dyspersji Incorez W2205 i nanonapełniaczy węglowych

Sample symbol	CNT		GNP	
	dispersion type	content [wt. part ^a]	dispersion type	content [wt. part ^a]
RS-0	—	0	—	0
A-0.5	Aquacyl	0.5	—	0
A-0.8		0.8		0
A-1.0		1.0		0
A-1.5		1.5		0
A/GNP-0.8/0.2	Aquacyl	0.2	ZUT-GNP	0.2
A/GNP-0.6/0.4		0.4		0.4
A/GNP-0.4/0.6		0.6		0.6
A/GNP-0.2/0.8		0.8		0.8
Z-0.5	ZUT-CNT	0.5	—	0
Z-0.8		0.8		0
Z-1.0		1.0		0
Z-1.5		1.5		0
Z/GNP-0.8/0.2	ZUT-CNT	0.2	ZUT-GNP	0.2
Z/GNP-0.6/0.4		0.4		0.4
Z/GNP-0.4/0.6		0.6		0.6
Z/GNP-0.2/0.8		0.8		0.8

a – wt. part/100 wt. parts of dry coat

Table 2. Surface resistivity and hardness of polyurethane coats with carbon nanofillers

Tabela 2. Rezystywność powierzchniowa i twardość powłok poliuretanowych z nanonapełniaczami węglowymi

Sample symbol	Nanofiller content [wt. part ^a]	Surface resistivity [Ω]	Hardness [a.u.]	Transparency [%]
RS-0	0 ^b	$9.0 \cdot 10^{14}$	100 ± 1	89
A-0.5	0.5	$1.0 \cdot 10^{10}$	100 ± 1	– ^d
A-0.8	0.8	$3.0 \cdot 10^8$	101 ± 1	38
A-1.0	1	$5.3 \cdot 10^7$	100 ± 1	35
A-1.5	1.5	$1.7 \cdot 10^7$	105 ± 1	23
A/GNP-0.8/0.2	0.8 ^b / 0.2 ^c	$2.7 \cdot 10^7$	103 ± 1	38
A/GNP-0.6/0.4	0.6 / 0.4	$4.1 \cdot 10^7$	106 ± 1	39
A/GNP-0.4/0.6	0.4 / 0.6	$1.2 \cdot 10^{10}$	100 ± 1	– ^d
A/GNP-0.2/0.8	0.2 / 0.8	$2.1 \cdot 10^{14}$	104 ± 1	– ^d
Z-0.5	0.5	$3.6 \cdot 10^{12}$	105 ± 1	– ^d
Z-0.8	0.8	$2.4 \cdot 10^7$	109 ± 1	38
Z-1.0	1	$7.0 \cdot 10^6$	101 ± 1	37
Z-1.5	1.5	$1.0 \cdot 10^6$	101 ± 1	– ^d
Z/GNP-0.8/0.2	0.8 / 0.2	$1.5 \cdot 10^7$	102 ± 2	38
Z/GNP-0.6/0.4	0.6 / 0.4	$7.3 \cdot 10^7$	101 ± 1	39
Z/GNP-0.4/0.6	0.4 / 0.6	$2.4 \cdot 10^{12}$	104 ± 1	– ^d
Z/GNP-0.2/0.8	0.2 / 0.8	$1.3 \cdot 10^{13}$	103 ± 1	– ^d

a – wt. part/100 wt. parts of dry coat; b – CNT content; c – GNP content; d – not tested

Table 3. Glass transition temperature values and thermal stability of polyurethane coats with carbon nanofillers

Tabela 3. Temperatury zeszklenia oraz stabilność termiczna powłok poliuretanowych z nanonapełniaczami węglowymi

Sample symbol	Nanofiller content [wt. part ^a]	T _g ^d [°C]	T _g ^e [°C]	T ₅ ^f [°C]	T ₁₀ ^g [°C]
RS-0	0 ^b	15.7	32.4	107.2	169.8
Z-0.5	0.5	14.7	31.0	95.3	211.2
Z-1.0	1.0	15.3	34.2	119.0	208.3
Z-1.5	1.5	15.3	33.8	138.8	230.4
Z/GNP-0.8/0.2	0.8 ^b / 0.2 ^c	15.5	33.4	132.3	228.7
Z/GNP-0.6/0.4	0.6 / 0.4	15.0	36.5	142.5	232.9
Z/GNP-0.4/0.6	0.4 / 0.6	14.6	32.3	180.4	234.8
Z/GNP-0.2/0.8	0.2 / 0.8	14.3	32.7	191.8	239.6

a – wt. part/100 wt. parts of dry coat; b – CNT content; c – GNP content;
d – determined using DSC technique; e – determined using DMA technique;
f – temperature at 5 % mass loss; g – temperature at 10 % mass loss

FIGURES

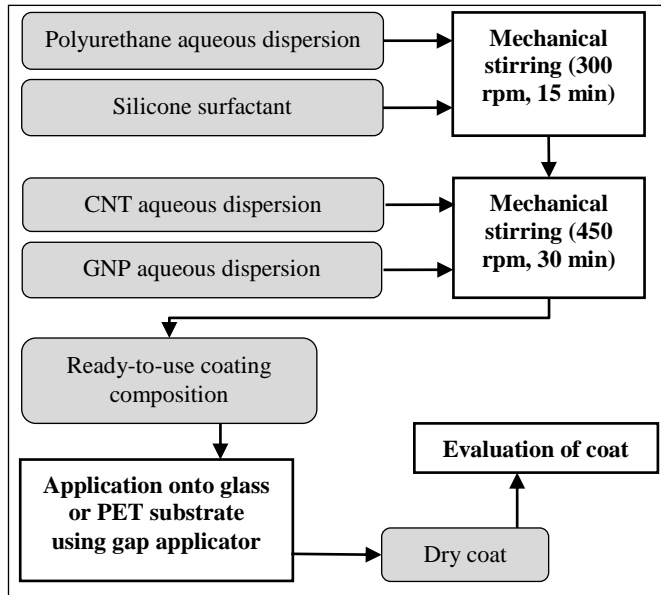


Fig. 1. Formulation scheme of polyurethane coating compositions and coats with carbon nanofillers

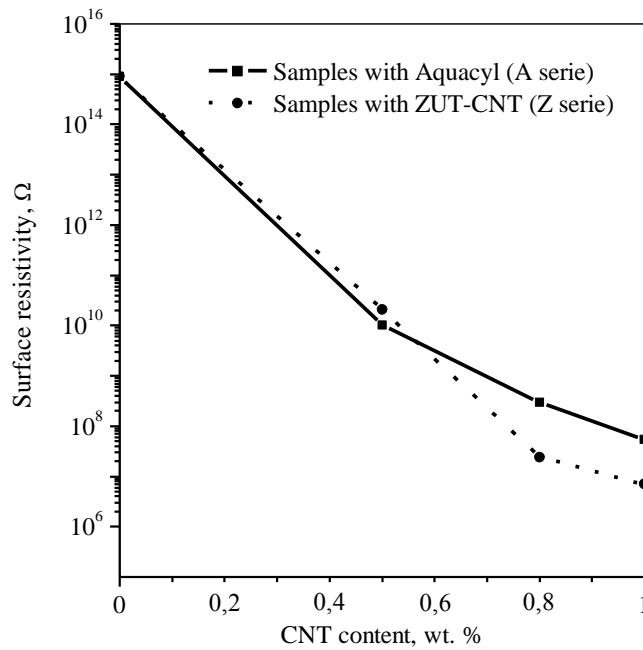


Fig. 2. Electrical surface resistivity of polyurethane coats with CNT (Aquacyl or ZUT-CNT)

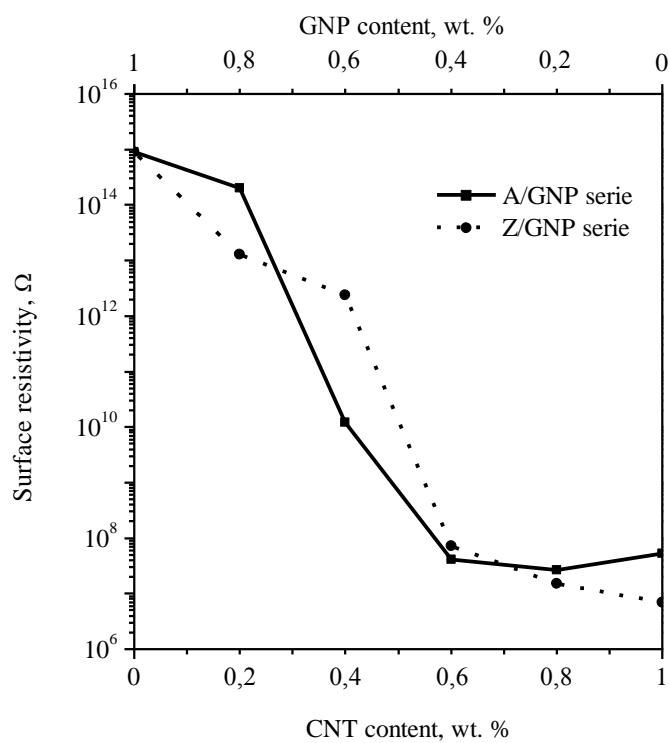


Fig. 3. Electrical surface resistivity of polyurethane coats with CNT/GNP mixtures (A is for Aquacyl, Z is for ZUT)

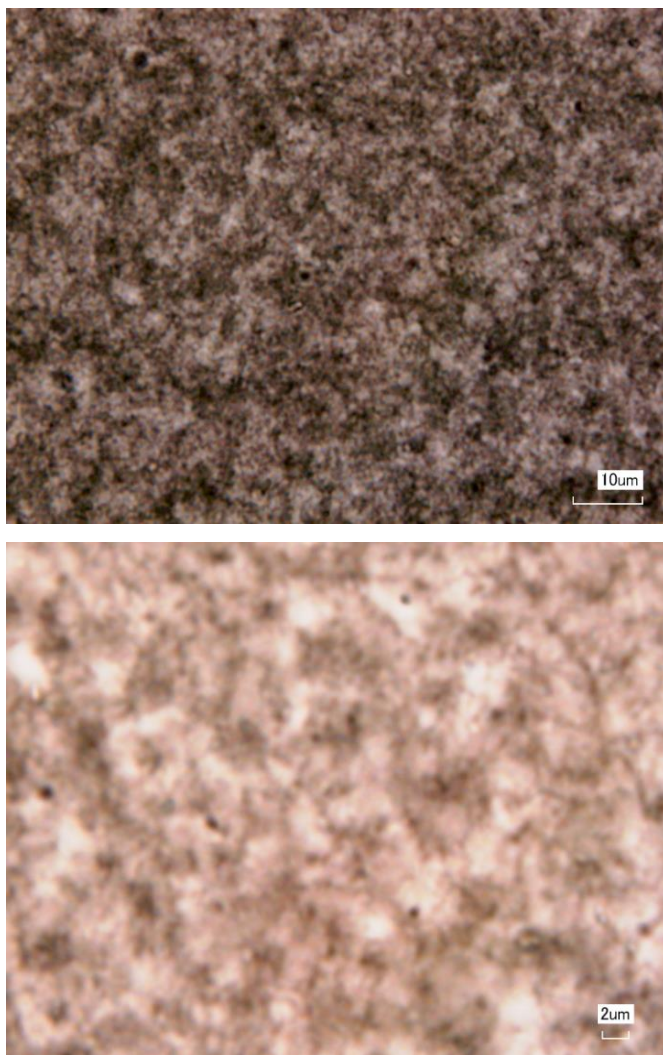


Fig. 4. LSM micrographs of polyurethane coat with CNT/GNP mixture (Z/GNP-0.6/0.4) at different magnification

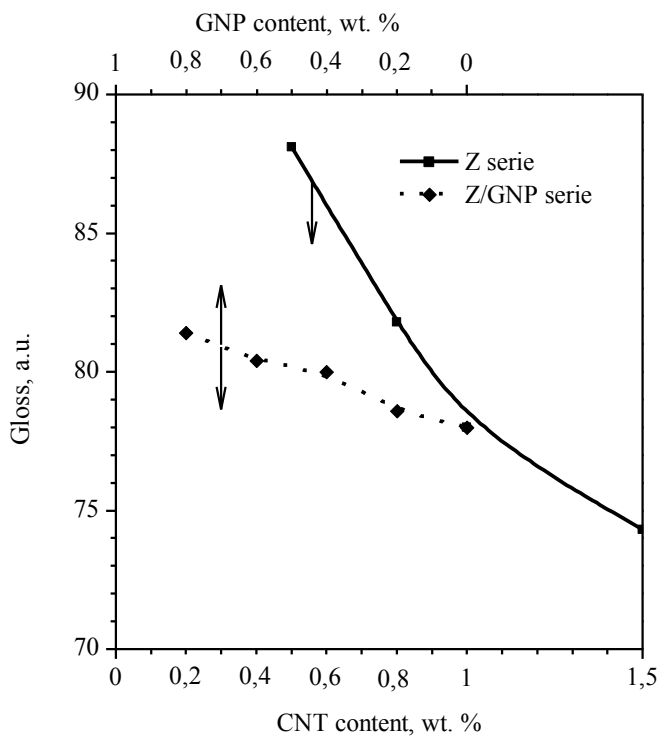


Fig. 5. Gloss of polyurethane coats with CNT and CNT/GNP

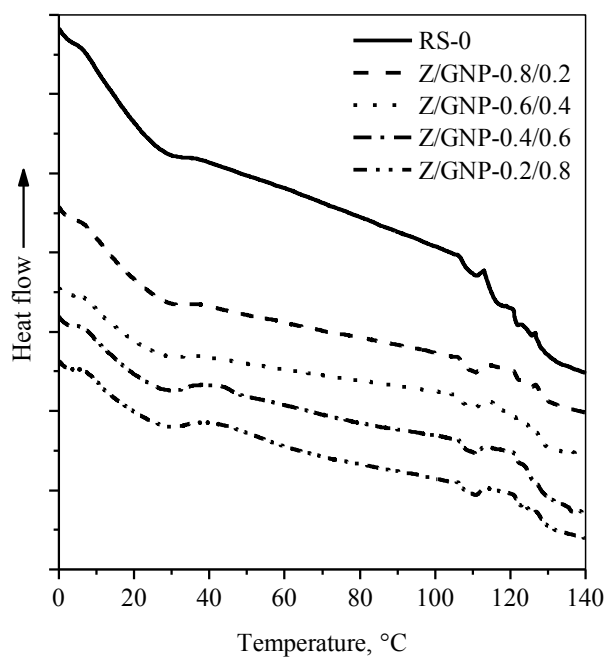


Fig. 6. DSC thermograms of polyurethane coats with CNT/GNP nanofillers mixtures

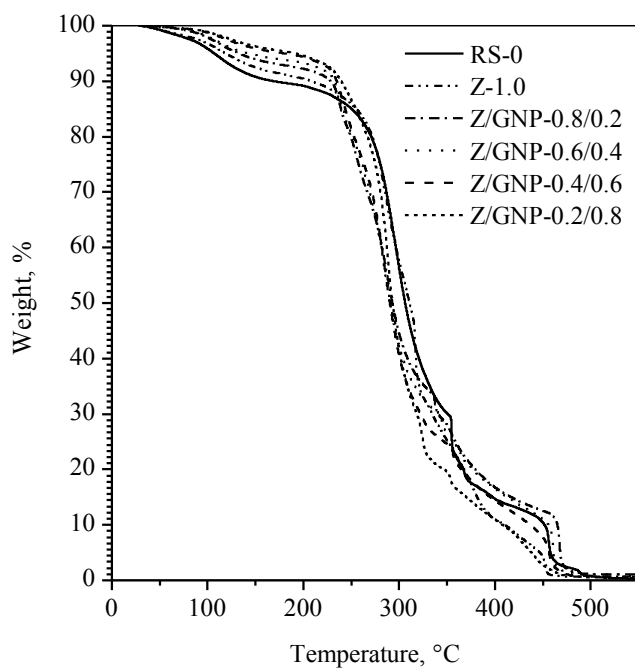


Fig. 7. TGA curves for polyurethane coats with CNT and CNT/GNP