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STATISTICAL ANALYSIS OF TRIBOLOGICAL STUDY RESULTS OF DRY ABRASIVE WEAR OF ULTRA-HIGH-MOLECULAR-WEIGHT POLYETHYLENE (UHMWPE), MODIFIED WITH CARBON NANOTUBES (CNTS)

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Abstract: *This arcticle deals with the statistical analysis of tribological results of Ultra-high-molecular-weight polyethylene (UHMWPE) samples containing different concentrations of multi-walled carbon nanotubes (CNTs) - 0.5%, 0.75%, 1.0% and 1.5% in several instances of contact interaction with the counterpart in the case of dry abrasive wear.*
Keywords: *abrasive wear, UHMWPE, carbon nanotubes, composite materials, statistical analysis*

1. INTRODUCTION

UHMWPE in its pure form has some very good antifriction properties - very good wear-resistance (less than 10^{15} J/m^3) which makes it superior to most of the composite polymeric materials used and it also has an extremely high resistance to erosion and abrasion. However, its coefficient of friction on metal for pressures 0,5÷5,0 MPa and sliding speeds 0,1÷2,0 m/s varies from 0,10÷0,35, which does not meet the requirements for good anti-friction material. This means that this material has to be modified to reduce its friction of coefficient, improve its thermal conductivity and mechanical strength. Carbon Nanotubes (CNTs) are allotropes of carbon with a cylindrical nanostructure which have unusual properties valuable for nanotechnology, electronics, optics and other fields of materials science and technology. Owing to the material's exceptional strength and stiffness, nanotubes have been constructed with length-to-diameter ratio of up to 132,000,000:1, significantly larger than for any other material. Their biggest advantages are that they are extremely small and light, resistant to temperature changes and self-lubricating. The Carbon Nanotubes used in the study are with an average diameter of 10÷40 nm, length of 1÷25 μm, purity by weight 93% and specific surface 150÷250 m^2/g. In order to improve their reinforcement properties, a surface electroless nickel plating (Ni-P) on SiC particles in alkaline bath using two nickel salts ($NiSO_4$ and $NiCl_2$) is conducted [3]. The main idea is to show the results from the R&D of a new composite material based on UHMWPE reinforced with CNTs, which in turn are reinforced with nickel plating, in order to combine the positive characteristics of both materials and to overcome each other's disadvantages to create a unique material possessing low coefficient of friction and high wear-resistance, which characteristics are retained in a wide range of working environments. The wear behavior test methodology used is based on the measurement of the mass wear of the specimens for a defined friction path under constant conditions - load,

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sliding velocity, type of abrasive and sample sizes, after which the wear characteristics are calculated - wear rate, wear intensity, absolute and relative wear-resistance.

The methodology includes the following operations:

- Preparation of samples of the same size and uniform roughness of the contact surface. The surface roughness and profile is determined by the roughness gauge "*TESA Rugosurf 10-10G*";
- Measurement of the starting mass m_o [mg] of the specimen using an electronic balance WPS 180/C/2 with an accuracy of 0,1. Before each measurement of the balance the sample is cleaned from any mechanical and organic particles and dried with ethyl alcohol to prevent the electrostatic effect;
- The sample is placed in the holder of the head of the tribotester (tribometer), a specified normal load P is assigned, and a specific path of friction S is realized.
- The mass m_i of the specimen is measured again after passing the specified friction path S.

The following characteristics of mass wear are calculated:

- *Mass wear m,* [mg] - represents the mass of the surface layer of the sample for a specific friction path S;
- *Mass wear rate* γ [mg/min] - it represents the removed mass friction per unit time of friction t;
- *Wear intensity,* i [μm/m] or [m/m] - the change of the height of the sample (linear wear) per unit of friction path S;
- *Wear-Resistance I* (absolute wear resistance) - it is expressed as a reciprocal value of the wear intensity *i*;

Absolute wear-resistance is a number that shows how many meters of friction path the specimen will pass under given friction conditions to destroy its surface area, equivalent to linear wear (h) a micron.

- *Relative wear-resistance* $R_{i,j}$ - represents the ratio of the absolute wear resistance of the test specimen (I_i) and the wear resistance of a test specimen adopted as a benchmark (I_j), determined under the same friction conditions;

Relative wear resistance is a non-dimensional number that indicates how many times the wear resistance of the test specimen is greater or less than that of the benchmark. The abrasive wear in dry friction is investigated with a kinematic "*Thumb-Disc*" kinematic device in flat contact.

The functional diagram of the device is shown in Figure1 [1,2]. The test specimen 1 (thumb) is fixed to the holder seat 2 in a loading head 8, in such a way that the face of the test specimen contacts the abrading surface 3, fixed for horizontal disc 4. The disc 4 is driven by an electric motor 6 and rotates about its vertical center axis with an angular velocity ω=const.

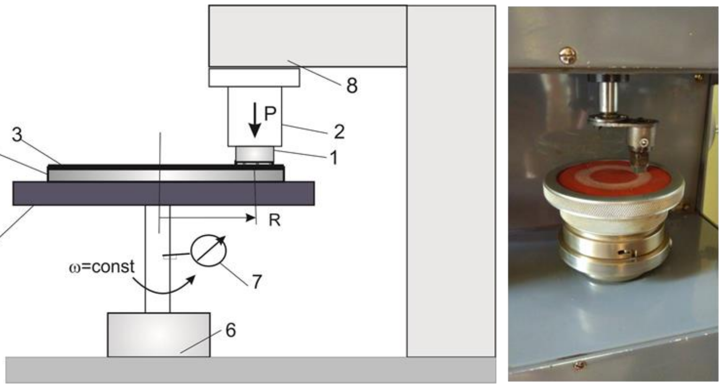


Fig.1. *Diagram of an apparatus for the study of the wear by friction on a surface with fixed abrasive scheme «Thumb-disk»*

The normal load P is applied to the center of gravity of the contact site between the sample and the abrasive surface and is provided with weights by means of a lever system in the loading head. The friction path is set by the number of revolutions with the tachometer 7. The device allows the sliding speed to be varied by varying the angular speed of the disc from the control unit and/or by varying the distance ***R*** between the axis of rotation of the disc 4 and the axis of the sample 1. The abrading surface 3 is molded by impregnated corundum (E) with a hardness of 9.0 on the Moos scale, which guarantees the requirement of the standard (БДС/ISO 14289:1977 and GOST 17 367-71) for a minimum of 60% higher abrasion hardness than that of the surface layer of test materials. The experimental study was conducted under the conditions shown in Table 1.

Table 1.

Experiment parameters

№	Parameter	Value
1	Load	P = 4.53 N
2	Nominal contact area	A_a = 144 mm^2
3	Nominal contact pressure	p_a= 3.14 N/cm^2
4	Speed of rotation	n = 200 min^{-1}
5	Distance between the axis of rotation and the center of the contact	R = 32 mm
6	Sliding speed of the center contact	V_C = 0.7 m/s
7	Abrasive surface	Corundum P320
8	Ambient temperature	T = 23°C

Table 2, Table 3 and Table 4 show the experimental results for mass wearing (mg), speed (mg/min) and mass wear intensity (m/m) for several cycles N = 100, 200, 300 and 400 respectively. The friction path S corresponding to the number of cycles N is calculated by the formula $S = 2\pi RN$ [m]. The designation of the tested materials UHMWPE include a figure indicating the corresponding percentage of carbon nanotubes CNTs [4,5]. For example, the UHMWPE-0.5CNTs designation indicates the presence of 0.5% carbon nanotubes CNTs. Nickel-plated carbon nanotubes are denoted by the letters Ni after the number, for example the designation UHMWPE-0.5Ni-CNTs should be read as: "*Ultra-high molecular polyethylene containing 0.5% nickel-plated carbon nanotubes*".

Table 2.
Mass wear of the test specimens under a different friction path (number of cycles)

№	MATERIALS	Number of cycles (N)			
		100	200	300	400
		Friction Path (m)			
		20.1	40.2	60.3	80.4
		Mass wear (mg)			
1A	Tufnol	20.2	35.7	55.8	71.1
2A	UHMWPE-0CNTs	1.7	3.5	4.7	5.6
3A	UHMWPE-0CNTs*	1.3	2.9	4.4	5.7
4A	UHMWPE-0.5CNTs	1.8	3.1	4.5	5.4
5A	UHMWPE-0.75CNTs	1.7	3.2	4.1	5.4
6A	UHMWPE-1.0CNTs	1.3	3.7	4.3	5.0
7A	UHMWPE-1.5CNTs	1.9	2.9	4.1	5.0
8A	UHMWPE-0.5Ni-CNTs	2.1	3.0	4.0	4.7
9A	UHMWPE-1.0Ni-CNTs	2.0	3.0	4.2	5.2
10A	UHMWPE-1.5Ni-CNTs	2.3	5.0	6.0	6.7

Table 3.
Wear rate of the samples at different friction paths (number of cycles)

№	MATERIALS	Number of cycles (N)			
		100	200	300	400
		Friction Path (m)			
		20.1	40.2	60.3	80.4
		Wear rate (mg/min)			
1A	Tufnol	84.2	37.2	38.8	37.0
2A	UHMWPE-0CNTs	7.08	3.65	3.26	3.92
3A	UHMWPE-0CNTs*	5.42	3.02	3.06	2.97
4A	UHMWPE-0.5CNTs	7.50	3.23	3.13	2.81
5A	UHMWPE-0.75CNTs	7.08	3.33	2.85	2.81
6A	UHMWPE-1.0CNTs	5.42	3.85	2.99	2.60
7A	UHMWPE-1.5CNTs	7.92	3.02	2.85	2.60
8A	UHMWPE-0.5Ni-CNTs	8.75	3.13	2.78	2.45
9A	UHMWPE-1.0Ni-CNTs	8.33	3.13	2.92	2.71
10A	UHMWPE-1.5Ni-CNTs	9.58	5.21	4.17	4.49

Table 4.
Wear intensity of the samples at different friction paths (number of cycles)

№	MATERIALS	Number of cycles (N)			
		100	200	300	400
		Friction Path (m)			
		20.1	40.2	60.3	80.4
		Wear Intensity (m/m)			
1A	Tufnol	4.98×10^{-6}	4.4×10^{-6}	4.59×10^{-6}	4.39×10^{-6}
2A	UHMWPE-0CNTs	0.63×10^{-6}	0.64×10^{-6}	0.58×10^{-6}	0.51×10^{-6}
3A	UHMWPE-0CNTs*	0.48×10^{-6}	0.53×10^{-6}	0.54×10^{-6}	0.52×10^{-6}
4A	UHMWPE-0.5CNTs	0.66×10^{-6}	0.57×10^{-6}	0.55×10^{-6}	0.50×10^{-6}
5A	UHMWPE-0.75CNTs	0.63×10^{-6}	0.59×10^{-6}	0.50×10^{-6}	0.50×10^{-6}
6A	UHMWPE-1.0CNTs	0.48×10^{-6}	0.68×10^{-6}	0.53×10^{-6}	0.46×10^{-6}
7A	UHMWPE-1.5CNTs	0.70×10^{-6}	0.53×10^{-6}	0.50×10^{-6}	0.46×10^{-6}
8A	UHMWPE-0.5Ni-CNTs	0.77×10^{-6}	0.55×10^{-6}	0.49×10^{-6}	0.43×10^{-6}
9A	UHMWPE-1.0Ni-CNTs	0.74×10^{-6}	0.55×10^{-6}	0.52×10^{-6}	0.48×10^{-6}
10A	UHMWPE-1.5Ni-CNTs	0.85×10^{-6}	0.92×10^{-6}	0.74×10^{-6}	0.62×10^{-6}

Table 5 and Table 6 show results respectively for the absolute and relative wear-resistance of the test specimens.

Table 5.

Wear-resistance of samples at different friction paths (number of cycles)

№	MATERIALS	Number of Cycles (N)			
		100	200	300	400
		Friction path (m)			
		20.1	40.2	60.3	80.4
		Wear Resistance (m/m)			
1A	Tufnol	2.0×10^5	2.3×10^5	2.2×10^5	2.3×10^5
2A	UHMWPE-0CNTs	15.9×10^5	15.6×10^5	17.2×10^5	19.6×10^5
3A	UHMWPE-0CNTs*	20.8×10^5	18.9×10^5	18.5×10^5	19.2×10^5
4A	UHMWPE-0.5CNTs	15.1×10^5	17.5×10^5	18.1×10^5	20.0×10^5
5A	UHMWPE-0.75CNTs	15.9×10^5	16.9×10^5	20.0×10^5	20.0×10^5
6A	UHMWPE-1.0CNTs	20.8×10^5	14.7×10^5	18.9×10^5	21.7×10^5
7A	UHMWPE-1.5CNTs	14.2×10^5	18.9×10^5	20.0×10^5	21.7×10^5
8A	UHMWPE-0.5Ni-CNTs	13.0×10^5	18.2×10^5	20.4×10^5	23.3×10^5
9A	UHMWPE-1.0Ni-CNTs	13.5×10^5	18.2×10^5	19.2×10^5	20.8×10^5
10A	UHMWPE-1.5Ni-CNTs	11.8×10^5	10.9×10^5	13.5×10^5	16.1×10^5

Table 6.

Relative wear-resistance

№	MATERIALS	WEAR RESISTANCE (S=80.4 m)	Relative wear resistance $R_{i,j}$				
			Influence of nanoparticles on UHMWPE		Comparison UHMWPE and Tufnol		Comparison UHMWPE w/o Ni and w/ Ni
			w/o Ni	w/ Ni	w/o Ni	w/ Ni	
1A	Tufnol	2.3×10^5	-	-	$R_{1,1}=1$	$R_{1,1}=1$	-
2A	UHMWPE-0CNTs	19.6×10^5	-	-	$R_{2,1}=8.5$	-	-
3A	UHMWPE-0CNTs*	19.2×10^5	$R_{3,3}=1$	$R_{3,3}=1$	$R_{3,1}=8.4$	-	-
4A	UHMWPE-0.5CNTs	20.0×10^5	$R_{4,3}=1.04$	-	$R_{4,1}=8.7$	-	$R_{4,4}=1$
5A	UHMWPE-0.75CNTs	20.0×10^5	$R_{5,3}=1.04$	-	$R_{5,1}=8.7$	-	-
6A	UHMWPE-1.0CNTs	21.7×10^5	$R_{6,3}=1.1$	-	$R_{6,1}=9.4$	-	$R_{6,6}=1$
7A	UHMWPE-1.5CNTs	21.7×10^5	$R_{7,3}=1.1$	-	$R_{7,1}=9.4$	-	$R_{7,7}=1$
8A	UHMWPE-0.5Ni-CNTs	23.3×10^5	-	$R_{8,3}=1.2$	-	$R_{8,1}=10$	$R_{8,4}=1.2$
9A	UHMWPE-1.0Ni-CNTs	20.8×10^5	-	$R_{9,3}=1.1$	-	$R_{8,1}=9$	$R_{9,6}=0.9$
10A	UHMWPE-1.5Ni-CNTs	16.1×10^5	-	$R_{10,3}=0.8$	-	$R_{10,1}=7$	$R_{10,7}=0.7$

2. STATISTICAL ANALYSIS OF THE OBTAINED RESULTS

The following variables are introduced [9÷13]:

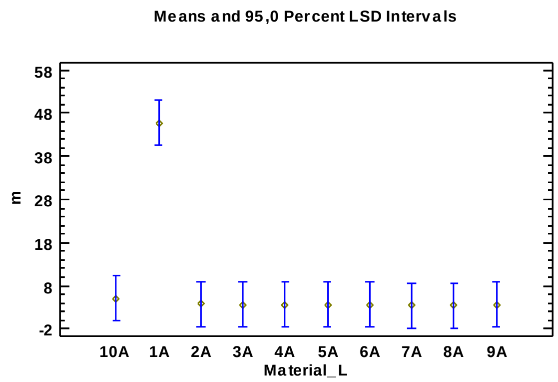
Variable	Description	Dimension
m	numerical variable: mass wear	[mg]
Gamma	numerical variable: wear rate	[mg/min]
i	numerical variable: wear intensity	[micro g/m]
II	numerical variable: wear-resistance	[m/micro g]
Rij_CN_UH_df	numerical variable: relative wear-resistance	[.]
Rij_UH_Tf_df	numerical variable: relative wear-resistance	[.]
Material_L	grouping variable (string)	
N	grouping variable: number of cycles (integer)	
	100 | 200 | 300 | 400	
S	grouping variable: friction path (integer)	
	20 | 40 | 60 | 80	
Ni_CN_UH	grouping variable: 0 – without Ni, 1 – with Ni	
Ni_UH_Tf	grouping variable: 0 – without Ni, 1 – with Ni	

Material_L – grouping variable (string)	Material	Material_1 – grouping variable (integer):
1A	Tufnol	1
2A	UHMWPE-0CNTs	2
3A	UHMWPE-0CNTs*	3
4A	UHMWPE-0.5CNTs	4
5A	UHMWPE-0.75CNTs	5
6A	UHMWPE-1.0CNTs	6
7A	UHMWPE-1.5CNTs	7
8A	UHMWPE-0.5Ni-CNTs	8
9A	UHMWPE-1.0Ni-CNTs	9
10A	UHMWPE-1.5Ni-CNTs	10

Each experimental magnitude is described by a random variable. Each factor which investigated is described by a grouping variable. Each numeric variable is considered separately.

One-way ANOVA: Analysis of the factor "material"

The factors are studied separately: material (wt% CNTs), number of cycles in the tests, distance. The dependent variable is mass wear. It is necessary to establish that the factors have a response to the dependent variable. According to Table 2, the 40 observations and the 10 levels of the factor "material" are examined. The mean values with their confidence intervals are shown in Figure 5 - Number of Observations: 40 and Number of Factor Levels: 10.



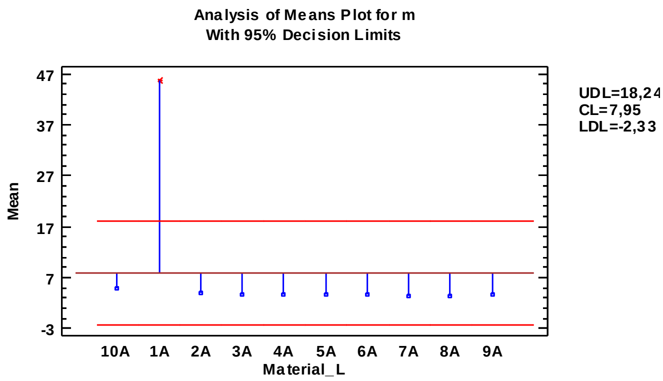


Fig.5 *Mean values and confidence intervals*

Fig.6 *Investigation of mean values and confidence limits*

The analysis of the mean values (Figure 6) shows the influence of the factor on the wear. Material 1A is sharply different from the rest (Figure 5 and Figure 6). If the data is censored and material 1A is dropped from the examination, the other materials in terms of wear can be represented by the following graph (Figure 7 and Figure 8). A response is observed with respect to material 10A.

Multi-factor ANOVA: Wear rate of the test samples

The co-influence of factors "material" and "number of cycles" on variable mass wear is examined by two-factor analysis. In the single factor analysis, the influence of the number of cycles factor is not clearly highlighted. The censored values are used. With respect to censorship of data, the same reasoning is used as the previous variable mass wear (Table 3). The wear rate is considered a random variable and it is necessary to determine the probability distribution.

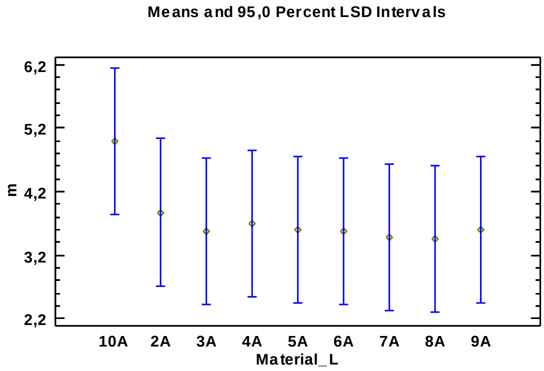


Fig.7. *Mean values of the censored data*

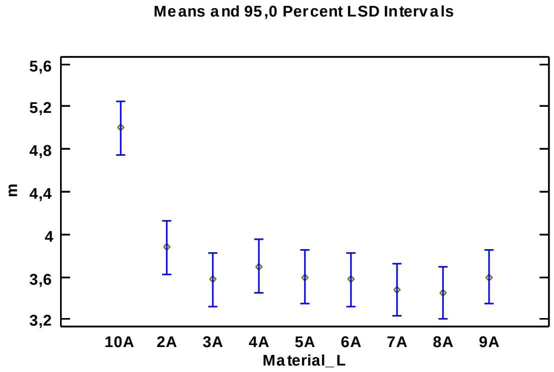


Fig.8. *Mean values with confidence limits*

One-way ANOVA: Analysis of the factor "material"

The factors are studied separately: material (wt% CNTs), number of test cycles, distance. The dependent variable is the wear rate. It is necessary to establish that the factors have a response to the dependent variable. According to Table 3, the 40 observations and the 10 levels of the factor "material" are examined. The mean values with their confidence intervals are shown in Figure 9 - Number of Observations: 40 and Number of Factor Levels: 10.

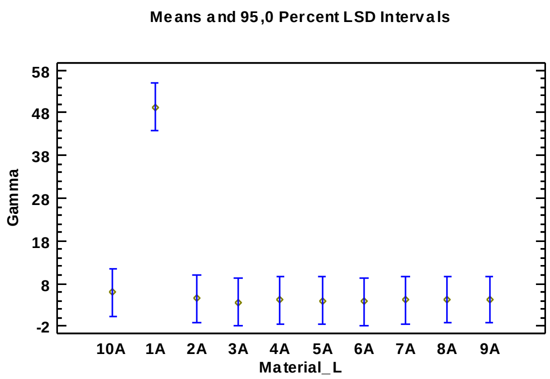


Fig.9. *Mean values and confidence intervals*

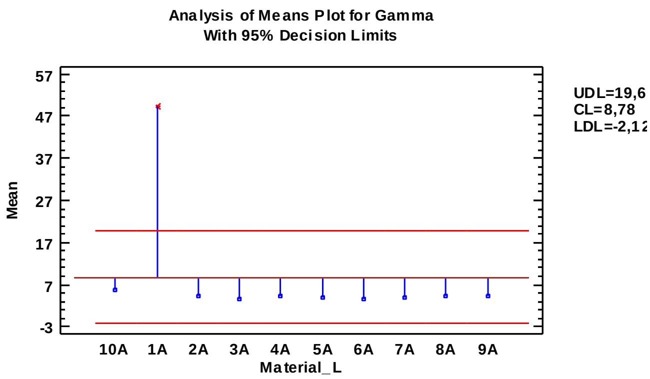
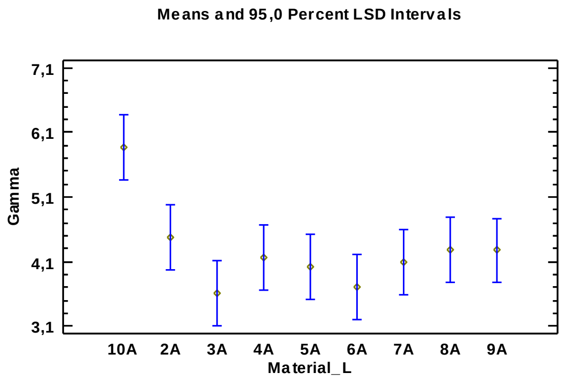


Fig.10. *Analysis of the mean values*

Material 1A (Tufnol) again has a substantial response to "wear rate" (Figure 9 and Figure10). To review the rest of the material in detail, this material can be censored. The censorship process can be interpreted as a re-iteration process when the data is viewed "under magnifying glass", i.e. a more detailed review of the results for materials 2A÷10A. If the factor “material” is censored, the response (influence) of the factor on the variable "wear velocity" is unclear. This is the reason to directly switch to two-factor data analysis.

Two-factor ANOVA: Wear intensity of the samples

The joint influence of factors "material" and "number of cycles" on the variable "wear rate" is examined by two-factor analysis. In the single factor analysis, the influence of the number of cycles factor is not clearly highlighted. The censored values are used.



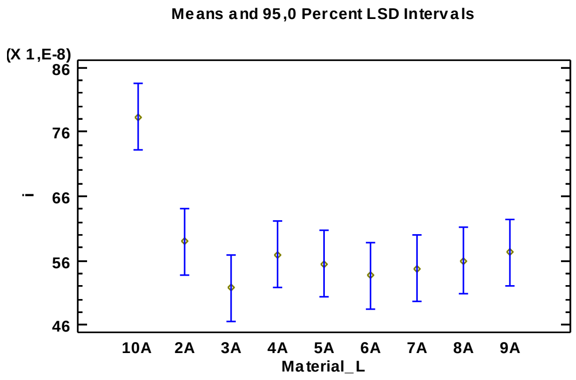


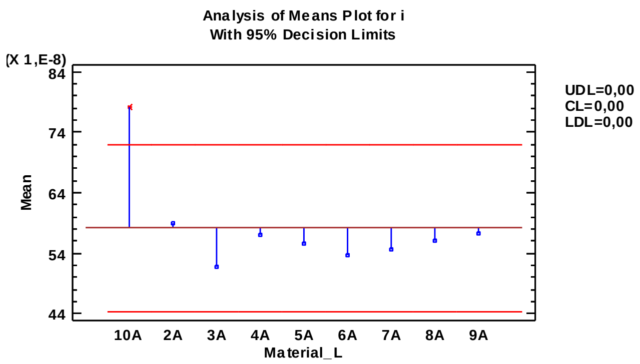
Fig.11. *Mean values and confidence limits for factor "material"*

Fig.12. *Mean values and confidence intervals*

With regard to censorship of data, the same reasoning as the previous variable mass wear can be carried out (Table 4). The wear intensity is considered a random variable and it is necessary to determine the probability distribution.

Two-factor ANOVA: Wear-resistance of the samples

The joint influence of factors "material" and "number of cycles" on the variable "wear intensity" is investigated by two-factor analysis. In the single factor analysis, the influence of the number of cycles factor is not clearly highlighted. The censored values are used. Both factors: "material" and "number of cycles" affect the accidental wear intensity. P-Values prove this conclusion.



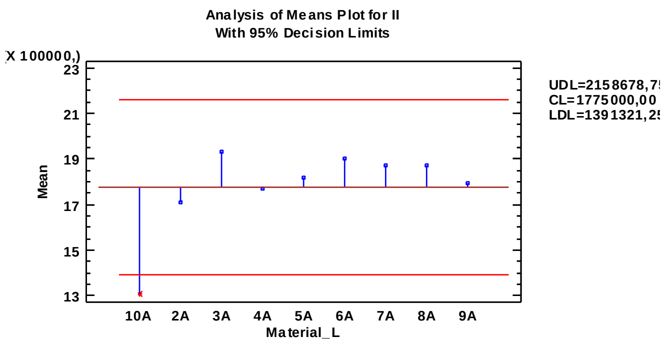


Fig.13. *Analysis of the response of mean values*

Fig.14. *Graphic representation of the mean values in terms of the factor "material"*

3. CONCLUSION

The dependence of friction wear on non-nanoparticulate UHMWPE [6] has a linear character. The presence of nanoparticles introduces a certain non-linearity in this dependence, which is more pronounced in nickel-plated nanoparticles. In nickel-plated nanoparticulate materials [7,8], the lack of treatment period was observed in a 1.5% nickel-plated specimen. The nonlinearities described in the kinetics of wear are confirmed by the change in wear intensity for non-nickel-coated specimens. Emphasizing the character of these curves is based on the fact that for abrasive wear, the generally accepted view amongst the tribologists is linear, proportional dependence of wear on the friction path. Figure 15 clearly shows the influence of the percentage of nanotubes with and without nickel coating on the abrasion resistance of UHMWPE materials in

400 friction cycles. The percentage of uncoated nanotubes has almost no impact on abrasion resistance in dry abrasive friction.

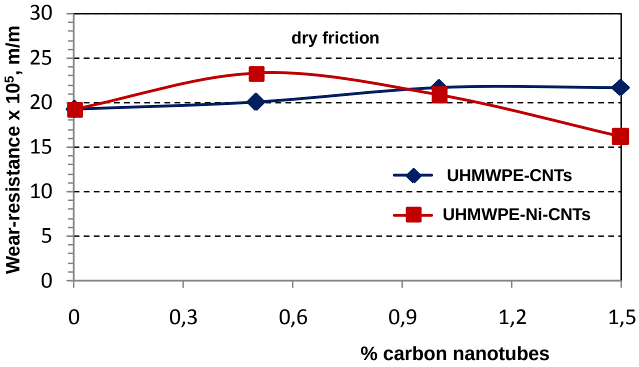


Fig.15. *Dependence of wear resistance from the percentage of nanoparticles for UHMWPE materials, with and without nickel coating*

For nanoparticulate coated materials, the dependence is nonlinear in nature with a pronounced maximum of wear-resistance in content 0.5% Ni-CNTs. With higher nanoparticle content, wear resistance decreases and for 1.5% it has the value $16.1x10^5$, which is smaller than that without nanoparticles - $19.6x10^5$ (Table 5). At this stage of the study it is assumed that the reduction of the wear resistance of the materials by the presence of 1.5% nickel-coated nanotubes in abrasive friction is due to an increase in the brittleness of the surface layer resulting in a decrease in the cohesion strength and consequently a decrease in its resistance against the cutting action of the abrasive particles. A diagrammatic representation of the absolute and relative wear-resistance of the samples is obtained from the diagrams presented in Figure 16 and Figure 17.

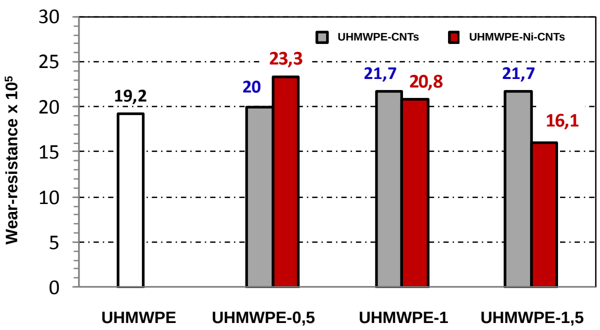


Fig.16. *Diagram of the wear-resistance of UHMWPE materials with carbon nanotubes with and without nickel plating for 400 cycles*

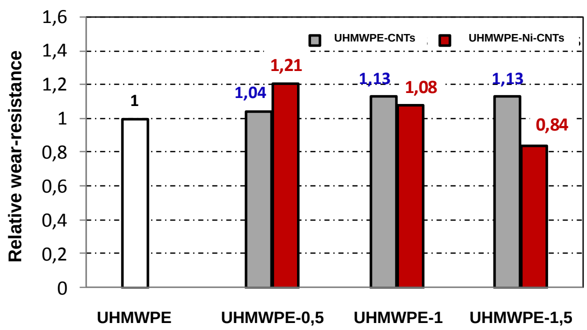


Fig.17. *Diagram of the relative wear-resistance of UHMWPE materials with carbon nanotubes with and without nickel plating for 400 cycles*

Wear-resistance is a reciprocal value of variable wear intensity. The results are identical with those obtained for "wear intensity". For the influence of the factors: "material", "friction time" and "friction path", the following conclusions can be drawn:

- The censored data is used to highlight the influence of factors on samples: 2A÷10A. The censorship considerations were clarified;

From the graphs presented in the statistical analysis it can be concluded that the best results for dry abrasive wear shows sample (8A) UHMWPE-0.5Ni-CNTs, which fully coincides with the results obtained from the tribological tests. The presented model fully justifies the experiment under consideration.

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