Arch. Metall. Mater., Vol. 61 (2016), No 4, p. 1881–1888

DOI: 10.1515/amm-2016-0303

M. OPALIŃSKI*,#, P. MAZURO *, A. KLASIK**, E. ROSTEK**

TRIBOLOGICAL EXAMINATION OF DIFFERENT STEEL MATERIALS AFTER SPECIAL HEAT TREATMENT AND SALT BATH NITRIDING

The main aim of the presented research was examination of new tribological pairs made of different types of steel. Materials of disc probes were submitted to unusual heat treatment processes and salt bath nitriding. The research is focused on the friction coefficients and mass losses of the material probes. Based on the results it was noticed that the best wear resistant pair was bearing steel 100Cr6 coupled with high speed steel S705. The lowest friction coefficient appeared for the pair bearing steel 100Cr6 and maraging steel C350.

Keywords: tribology, mechanical properties, steel alloys, friction coefficient

1. Introduction

Currently one of the most important parameter of engines is the efficiency. In the energetics and transportation the major attention is paid to the engine efficiency since this parameter determines the profitability of those businesses. There are two main groups of factors influencing the engine efficiency: mechanical and thermodynamic factors. In the first group the biggest impact has a construction of the engine and the way the piston forces are transferred on the engine shaft. Additional mechanical factors are the type and number of bearings, surface coatings of parts, lubrication and combustion chamber sealing. Thermodynamic factors are inter alia preparation of combustible mixture (either direct injection or carburetor solution), control and type of combustion, realization of the cooling system and turbocharging systems.

Recently a lot of interest is oriented on the opposed piston engines. Based on this construction there is a possibility to limit or even to eliminate the combustion chamber cooling and thus to limit the heat losses of the engine [1] bringing it closer to the adiabaticity.

Limiting the heat loses is directly connected with an increased temperature of the engine elements and more severe tribological conditions [2,3]. In regards to increased tribological requirements in particularly for the piston sealing ring and cylinder liner surface it was decided to carry out the lab tests for selected types of steel to explore completely new tribological pairs.

2. Aims of the research

The research was aimed at two aspects. The first aspect regarded finding the couple with the lowest friction coefficient. The latter aspect was connected with the lowest surface abrasive wear of contacting probes. Both aspects are correlated with the engine efficiency. The first one has impact on the losses caused by friction forces in the piston reciprocating motion due to side piston force. The second aspect is connected with gas leakages from the combustion chamber to the crankcase [4,6]. This influences the pressure drop in the combustion chamber usually implying the loss of engine power [3,8]. The main goal of the work was to propose new tribological pair for the piston sealing rings and cylinder liners, which could be applied in the opposed piston barrel engines. This type of engine is currently developed at the Institute of Heat Engineering, Warsaw University of Technology. For the research purposes investigated steel alloys were very diversified: austenitic steel, martensitic steel, heat-resistant steel, tool steel, valve steel etc. More details about the materials selected for test probes are presented in the section 3b. All alloys were submitted for unusual heat treatment and salt bath nitriding. The tests were carried out in the severe conditions simulating extreme piston cylinder working environment. During the test there was no lubrication (dry friction), the temperature in the test chamber was raised to 150oC and the contact pressure was 4MPa.

3. Test description

3.1. Lab bench

The research was carried out on a tribological tester T-11 type Pin-on-Disk which is intended for measuring the tribological parameters of materials at the contact node. The device can be used for different materials such as metals, plastics and ceramics in the environment of air in controlled temperature of up to 200°C [5]. The tester T-11 is presented in the Figure 1. The scheme and operating principle is shown in the Figure 2.

* WARSAW UNIVERSITY OF TECHNOLOGY, INSTITUTE OF HEAT ENGINEERING, 21/25 NOWOWIEJSKA STR., 00-665 WARSZAWA. POLAND

** MOTOR TRANSPORT INSTITUTE, 80 JAGIELLOŃSKA STR., 03-301 WARSZAWA. POLAND

Corresponding author: marcin.opalinski@itc.pw.edu.pl

Fig. 1. Tribological tester T-11 used in the laboratory tests

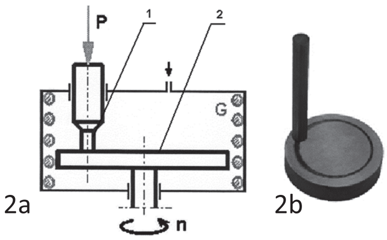

Fig. 2. Scheme (2a) and operating principle (2b) of the tribological tester

3.1.1 Working principle

Stable probe 1 which has a cylinder shape is pressed with force P to the rotating disk with the rotational velocity n (counter-probe). The node of friction is placed in the isolated chamber which is equipped with heating element G. The friction between the probe and counter-probe is transferred on the force sensor placed in the probe 1 mounting port. Based on that it is possible to measure the friction force and calculate the mean effective friction coefficient using equation below (1).

$$\mu = \frac{T}{P}, [-] \qquad (1)$$

where:
T – friction force [N]
P – node pressing force [N]

Based on the loss of mass of samples wear intensity factor (WIF) was calculated. The parameter describes the intensity of material loss at the contact node for selected friction path and contact area. The parameter is calculated according to the following equation (2) [5]:

$$I = \frac{M_1 - M_2}{SF}, \left[\frac{mg}{m^3}\right] \qquad (1)$$

where:
S – length of contact path [m]
M_1, M_2 – mass of probe before and after the test [mg]
F – contact area [m^2]

Tribological tests were conducted in the technical dry conditions [7] at temperature of 150°C. All test parameters are presented in the Table 1.

TABLE 1

Parameters and conditions of conducted test

Contact pressure	4 [MPa]
Test time	10000 [s]
Sliding speed	0,65 [m/s]
Loading applied to the pin	50 [N]
Length of friction path	6500 [m]
Rotating velocity of the disk	640 [obr/min]

For surface analysis of worn coating CONTOUR GT profilometer brand BRUKER was used. This is an optical type profilometer intended for analyzing the surface topography.

Optical profilometer (Figure 3) is based on the light interference phenomenon. The reflected light from the measured surface and the light reflected from the reference surface interfere and this results in the interference stripes. The image of interference stripes provides the information required to define the distance between measured surface points and microscope optical head.

Fig. 3 . Optical profilometer Contour GT Bruker

The tests can be done in accordance with norms: PN-EN ISO 4288, PN-EN ISO 4287, PN-EN ISO 25178-6 [5].

3.2. Selected materials

Tribological tests were conducted for the selected bearing needles (dimensions: radius 4 mm, length 39.8 mm) which were produced by Tsubaki Hoover company. The pin probes are made of bearing steel 100Cr6. The pins were coupled with the disc probes, which were made of the following materials:

- 1.4021 / X20Cr13 / 2H14 – martensitic stainless steel (Probe: Y 14)
- 1.4871 / X53CrMnNiN21-9 – valve austenitic steel (Probes: 5 71, 5 75)
- 1.4718 / X45CrSi9-3 – valve martensitic steel (Probe: 6 75)
- X2NiCoMoTi18-12-4 / C350 – maraging steel (Probe: 11 35)
- Uddeholm Corrax® – age hardened stainless steel (Probe: X 35) [13]
- 1.4841 / H25N20S2 – heat-resistant austenitic steel (Probes: 7 75, 7 71)
- Böhler M390 – powder stainless steel wear resistant

(Probe: T 74) [12]
- 1.4122 / X35CrMo17 / 3H17M – martensitic stainless steel (Probe: 4 75)
- Böhler K360 – tool steel for cold work (Probe: K 75) [12]
- 1.3243 / W6Mo5Cr4V2Co5 / S705 – high speed steel (Probe: I 74)

3.3. Material heat treatment

All materials were submitted for heat treatment. Depending on the steel type probes were age hardened or quenched and tempered in accordance to the Figure 4. At the beginning the probes were 4 level heated until they reached the hardening or quenching temperature. Then the materials were cooled and submitted for 3-level aging or tempering accordingly. After the heat treatment disc probes were grinded to the roughness 0.32 µm and then probes were submitted for salt bath nitriding. More details regarding temperatures of processes were presented in Table 2.

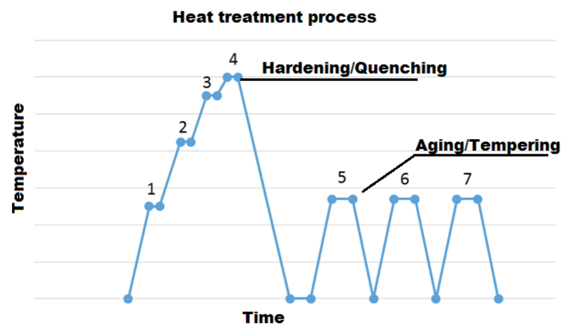

Fig. 4. Scheme of heat treatment. Steps 1-4 present 4-level heating to the temperature of hardening or quenching. Steps 5-7 present 3-level aging or tempering

3.4. Surface treatment

Probes after heat treatment were delivered to TS Zbąszynek hardening shop. Firstly the surface of probes was degreased. Next the probes were heated in air and submitted for salt bath nitriding. After the salt bath nitriding probes were cooled in water and then submitted for passive salt bath. The passive salt bath is intended for increasing the resistance to corrosion and gives anthracite black colour of the surface. In the next step the probes were cooled, cleaned and impregnated. The probes were submitted for three different nitriding cycles depending on the material:

1. 60 mins in temperature 580°C
2. 150 mins in temperature 530°C
3. 90 mins in temperature 580°C

Scheme presenting the whole process of nitriding is illustrated by Figure 5.

Additional tests and analysis of nitriding diffusion layer were performed during the engine project. Several probes were cut and their surfaces were etched (4 % nital) for the optical analysis. During the surface analysis it was noticed that the highest thickness of dark diffusion and layer of compounds was observed for the surface nitriding which lasted 90 mins in 580°C.

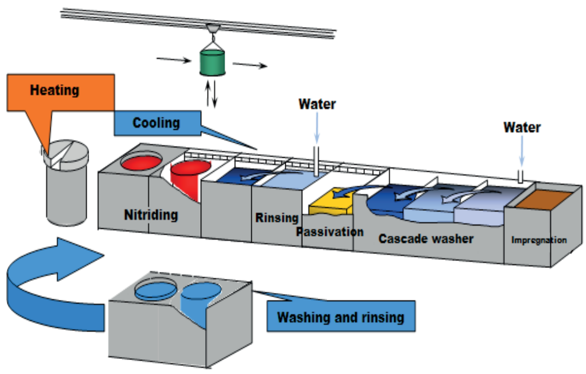

Fig. 5 . Scheme of nitriding treatment in TS Zbąszynek hardening shop [11]

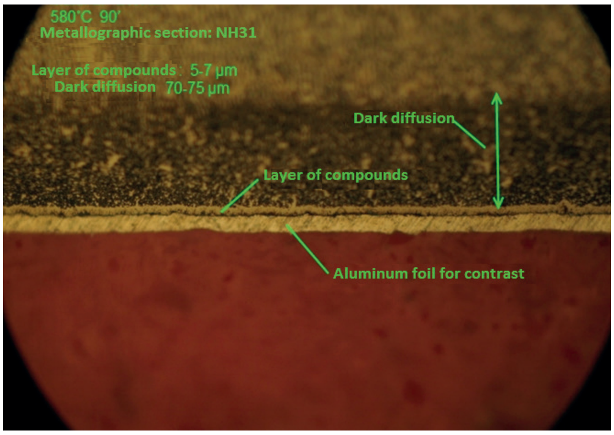

Fig. 6 . Cross section of nitriding layer of probe number 7 75

Example of probe surface analysis with description of layers is presented in Figure 6.

3.5. Summary of probes' treatments

All probes are compared in Table 2. The temperature of hardening or quenching was between 950-1150°C and the aging or tempering temperature was between 540-720°C. The probes were submitted mostly for 90 minutes for salt nitriding at temperature 580°C.

TABLE 2

Summary of materials and respective treatments

Probe number	Material	Quenching/ Hardening	Tempering/ Aging	Nitriding
Y 14	1.4021	1050	540	60min 580°C
5 71	1.4871	1150	720	150min 530°C
6 75	1.4718	1050	720	90min 580°C
11 35	C350	950	600	90min 580°C

X 35	Corrax	950	600	90min 580°C
7 75	1.4841	1150	720	90min 580°C
T 74	M390	1150	720	60min 580°C
4 75	1.4122	1050	720	90min 580°C
7 71	1.4841	1150	720	150min 530°C
K 75	K360	1050	720	90min 580°C
I 74	S705	1150	720	60min 580°C
5 75	1.4871	1150	720	90min 580°C

4. Research results

After each test of the tribological pair T-11 tester report was printed. The following results were saved on the disc:

- friction force
- displacement of pin mounting indicating total linear wear of tribological pair
- temperature of the probe.

All results were presented as a function of time. Any abrupt and sharp changes of those function indicated adhesive interaction between the materials. Smooth and continuous function was indicating proper tribological work.

Sample report of probe number 4 75 is presented in Figure 7. The report results indicates proper tribological work with final friction force on the level of 25 N and presents total tribological pair linear wear.

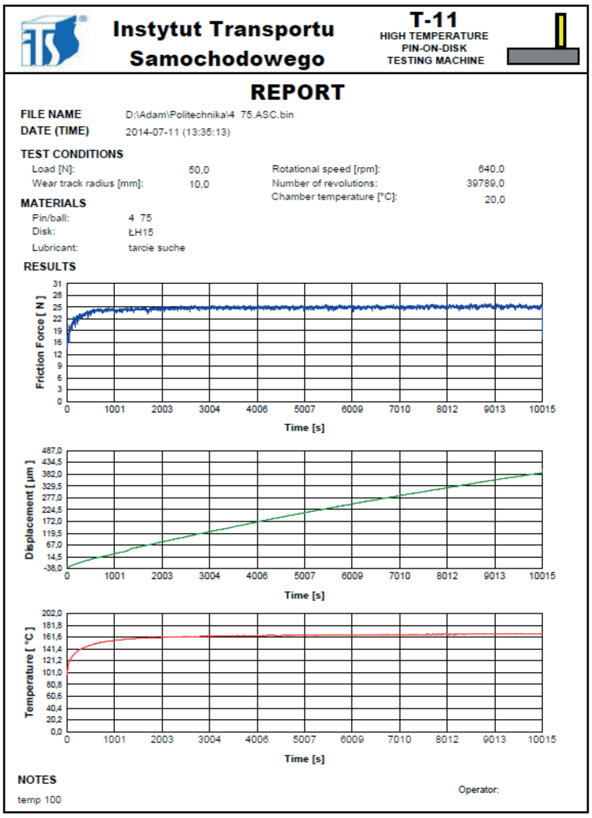

Fig. 7. Example of tribological test report of probe 4 75

Friction coefficients and WIFs, which were calculated according to the equations 1 and 2 were compared in Table 3.

TABLE 3

Friction coefficients and wear intensity factors for tribological pairs

Probe number	Frictionz coefficient µ	I disc [mg/m^3]	I pin [mg/m^3]
Y 14	0,53	36,75	210,68
5 71	0,50	17,15	414,01
6 75	0,54	64,92	334,39
11 35	0,44	56,34	334,39
X 35	0,47	90,64	309,90
7 75	0,51	6,12	329,50
T 74	0,51	15,92	284,17
4 75	0,48	25,72	314,80
7 71	0,58	4,89	432,39
K 75	0,51	17,14	703,09
I 74	0,55	75,94	241,30
5 75	0,54	20,82	400,54

Summary of all results from tribological tester T-11 were also illustrated in column graph (Figure 8). Single column presents total wear intensity factor (TWIF) for pin and disc. Disc intensity factor is the lighter part of each column. The dark part presents pin wear intensity factor. Additionally friction coefficients were added on the same graph to present whole tribological pair work.

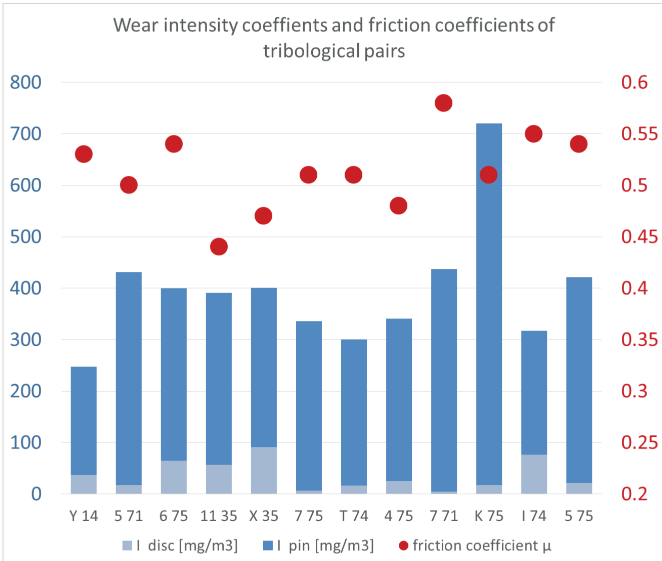

Fig. 8. Summary graph presenting total wear intensity factors of tribological pairs in connection with friction coefficients

Figure 9 presents the impact of materials submitted for the same surface (N) and heat treatments (HT). Materials presented by columns in the same colour were treated in the same way. Materials 1.4871 and 1.4841 were represented by four different probes after diverse surface nitriding. It may be noticed that the probes which were submitted for nitriding for 90 mins at 580°C presented lower wear than the probes nitriding at 530°C for 150 mins.

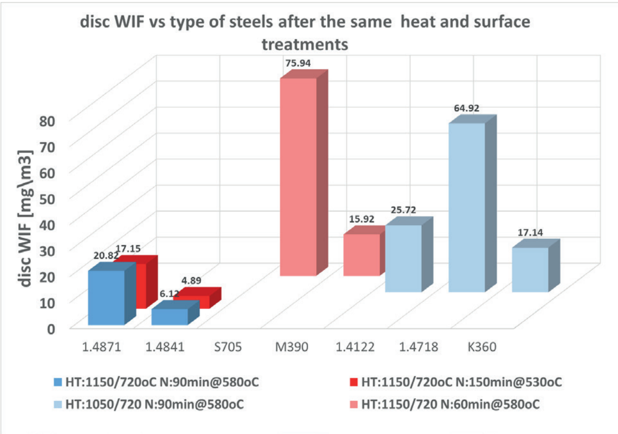

Fig. 9 .Materials after selected heat treatments (HT) and surface nitriding process (N) compared in regards to the same probes treatments (columns in the same colours)

Observations of the reports indicated similar type of friction for tribological pairs. Most of the materials (probes: 4 75, 6 75, 7 75, 5 71, 11 35, K 75, T 74, Y14) presented very smooth working conditions for the whole test. Friction force oscillations, after run-in time were between ±2.5% and ±5.5% of the total friction force. The rest materials (I 74 5 75, 7 71, X 35) presented more adhesive behavior. Figure 10a illustrates smooth work for probe T 74 presenting working conditions of the first group. Figures 10b and 10c are presenting the second group of materials. That group indicated more severe tribological work. Adhesive joints, which were appearing during the tests caused higher force amplitudes. For instance friction force for probe X 35 (Figure 10b) varied from 14 N to 39N for the time range 1800-3700 s, what gives (for the friction force of 24 N) force oscillations between -41.7 % and +62.5 %.

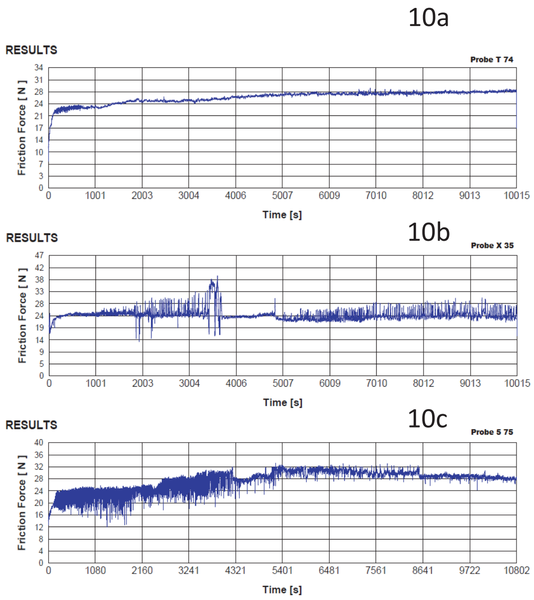

Fig. 10 .Friction force of 3 selected probes presenting different types of interaction

Observations of the surfaces of disc probes after the tribological tests indicated differential mechanisms of wear. Mass loss of the probes was caused by scratching, ridging or micro-abrasion. The differences between probes were connected with several factors mostly related to the type of material but also with parameters of surface and heat treatments.

Besides analysis of the data from tests which were conducted on tribological tester T-11 surface topology was also examined using the laboratory stand ContourGT. Results of surface topology indicated following effects of tribological pairs work:

- equality of wear and shape of the friction path,
- surface roughness.

Results of one probe (6 75) were illustrated in Figure 11. Two perpendicular profile lines located on the disc probe surface are showing the profiles of wear path in the radial and longitudinal direction. The profiles are supported with 3D picture of the surface.

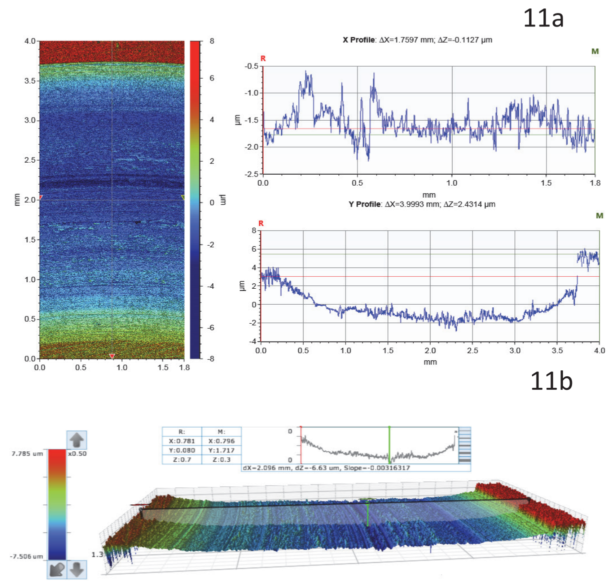

Fig. 11.Results of topographical measurements for probe 6 75. Figure 10a – view of the surface of disc with marked lines of surface profiles. Figure 10b – 3 dimensional image of worn surface

Table 4 presents the results which were gathered during measurements of the surface after tribological measurements.

TABLE 4

Results of tribological measurements of friction pairs after the tests

Probe number	Sa [μm]	Sq [μm]	Sv [μm]	Sz [μm]
Y 14	1,2	1,55	-31,56	39,03
5 71	0,57	0,71	-8,01	16,75
6 75	1,46	1,94	-7,51	15,29
11 35	1,89	2,15	-5,19	1,47
X 35	3,28	3,85	-8,83	19,78
7 75	0,62	0,8	-5,19	9,16
T 74	0,78	0,91	-5,18	11,38
4 75	1,5	1,76	-3,65	12,91
7 71	0,45	0,56	-7,33	13,46
K 75	1,52	1,82	-7,19	16,67
I 74	1,19	1,64	-6,45	13,63
5 75	0,99	1,39	-8,33	15

Topographic parameters of disc probes after the frictional tests were described below:
Sa – mean arithmetic deviation of average surface which is the arithmetic average of absolute values of deviations of average surface;
Sq – mean square deviation of surface described as standard deviation of surface height inequality measured from relative surface.
Sv – maximal depth of surface
Sz – the biggest height of roughness described as topographical height based on 10 points [9].

Results of topography analysis are presented in Figures 12 and 13. The biggest difference between highest point and the deepest point (Sz-Sv) presents probe Y 14 while the smallest probe 11 35. Small surface deviations (below 1µm) are represented by probes 5 71, 7 75, T 74, 7 71. The largest surface deviations was noticed for probe X 35.

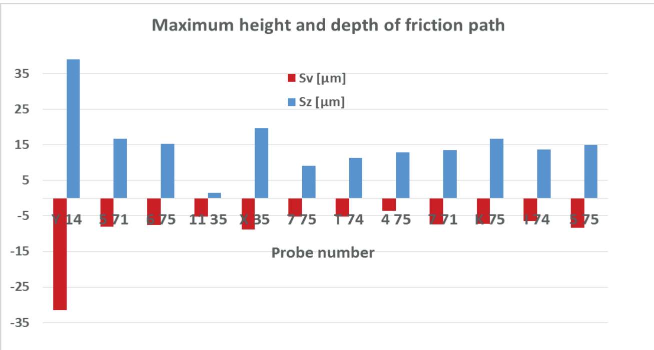

Fig. 12.Maximum height Sz and depth Sv of inequality of the probes' surfaces after tribological tests [µm]

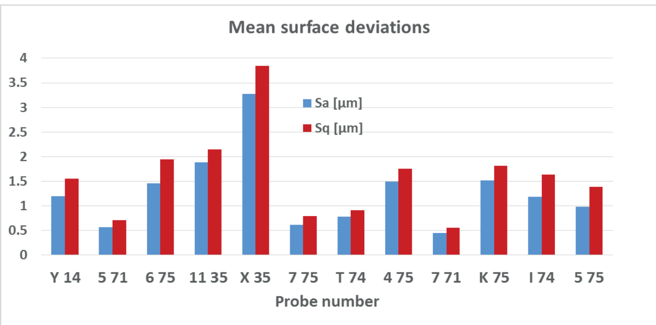

Fig. 13 .Mean square deviation and mean arithmetic deviation of the probes' surfaces after tribological tests

5. Conclusions

Three dimensional images of friction nodes indicate that the wear mechanism for the probes was different. The probes before tests were grinded to the same roughness of 0.32 µm. After the tests the mean arithmetic deviation differed from 0.5 to over 3 µm.

Friction coefficients were in the range of 0.44÷0.58 The lowest friction coefficient was noticed for 11 35 probe while the highest for 7 71 probe. Average friction coefficient was 0.51

TWIFs ($I_{disc}+I_{pin}$) were between 247.43 and 720.23 mg/m³. Probe Y 14 presented the lowest and K 75 the highest TWIF.

Average WIF for pin probes which were made of bearing steel (100Cr6) was 333.92 mg/m³. For disc probes average WIF was 47.77 mg/m³ which is 7 times less. This is mostly caused by surface treatment of the disc probes.

There is no apparent correlation between friction coefficient and wear intensification factor.

Probes 7 75 and 7 71 represented material H25N20S2 after the same heat treatment and different surface nitriding. Disc probe 7 75 was salt bath nitrided in 580°C for 90 mins while 7 71 in 530°C for 150 mins. The probe 7 71 due to longer nitriding presented lower wear. WIF for this probe was 4.89 mg/m³. This was 25.2% lower than for probe 7 75 which WIF was 6.12 mg/m³. Lower disc wear caused higher pin wear by 23.8%. WIF for disc probe 7 71 was 432.39 mg/m³ while for 7 75 was 329.5 mg/m³. TWIF for tribological pairs 7 71 and 7 75 was higher for the pair with longer nitriding. Similar behavior was presented by probes 5 71 (150' 530°C nitriding) and 5 75 (90' 580°C nitriding) which were representing 1.4871 valve steel. For longer nitriding disc probe wear was lower by 21.4% (17.15 mg/m³ and 20.82 mg/m³) while pin probe wear was higher by only 3.2% (414.01 mg/m³ and 400.54 mg/m³). TWIF for those probes was also higher for the pair with longer nitriding but not as much as for the previous probes. This observations should be taken into consideration while deciding what parameters of nitriding process should be set to obtain well balanced cylinder liner and piston ring wear.

Comparison of austenitic steels (probes 5 71, 7 71, 7 75) and martensitic steels (probes Y 14, 4 75, 6 75) indicate slightly better tribological performance of martensitic steels. Austenitic steels provides friction coefficient in the range of 0.5÷0.58 and WIF in the range of 329.5÷432.4 mg/m³ while martensitic steels are in ranges 0.48÷0.54 (FC) and 210.7÷334.4 mg/m³ (WIF).

Analysis of chemical composition of steels does not indicate direct influence for the tribological pairs. The structures which are appearing in the steels are more complex than separate compounds and it may vary for the same steel probes for different heat treatments. Results may be also influenced by different parameters of surface treatments. Table 5 presents chemical composition of 4 disc probes which are representing 2 most worn and 2 least worn tribological pairs with resulting friction coefficients.

TABLE 5

Tribological properties with middle range steel chemical composition for two most worn and two least worn pairs

Probe	Material	µ	Idisc+Ipin	C	Si	Mn	Cr	Mo	Ni	W	V
Y 14	1.4021	0.53	247.43	0.21	0.5	0.8	13	0	0	0	0
T 74	M390	0.51	300.09	1.9	0.7	0.3	20	1	0	0	0
7 71	1.4841	0.58	437.28	0.01	2.25	0	25	0	20.5	0.6	4
K 75	K360	0.51	720.23	1.25	0.9	0.4	8.8	2.7	0	0	1.18

All results are indicating that the tribological properties are very complex. Surface treatment and heat treatment with connection to material composition are making very difficult to predict and propose best tribological pairs. Many factors need to be taken into consideration before selecting most adequate tribological pair. Cylinder liner and piston ring materials should satisfy following requirements:

smooth, regular surface wear, low friction coefficients, serviceability, sealing properties. The best tribological disc probe in regards to the lowest wear intensity factor is Y 14 although from serviceability point of view T 74 may be considered better since it has also low TWIF but the pin probe for this material is more prone to wearing. Pin is representing material of piston rings which are much easier to replace then the cylinder liner. The materials for the typical piston rings are very diversified (various steel alloys and cast-iron) [14] and during the engine project several materials will be tested as a piston sealing rings. From the engine efficiency point of view well balanced relation between friction coefficient (0.44) and wear intensity factor (390.73) provides probe 11 35 (maraging steel C350). This probe had also the lowest mean surface deviations after tests what is valuable for sealing purposes.

Selecting proper tribological materials requires analyzing also other parameters such as heat resistance, creep resistance, thermal expansion coefficients and structural strength. Every engine design, application and type of part couplings have to be analyzed separately to select the best matching tribological pair.

Acknowledgements

Presented work is a part of the research which received funding from the Polish-Norwegian Research Program operated by the National Centre of Research and Development under the Norwegian Financial Mechanism 2009-20014 in the frame of Project Contract No Pol-Nor/199058/94

REFERENCES

[1] M. Szczeciński, P. Mazuro, W. Skrobek, Scavenging process CFD computations in a two stroke opposed-piston engine, PTNSS–2015–3404, **563** (2015).

[2] J-P. Pirault, M. Flint, Opposed piston engines: evolution, use, and future applications, SAE International, **p6**, Warrendale 2010.

[3] W. Kozaczewski, Konstrukcja grupy tłokowo-cylindrowej silników spalinowych, WKiŁ, **7-19**, Warszawa 2004.

[4] J. Włodarski, Tłokowe silniki spalinowe – procesy trybologiczne, WKiŁ, **29**, Warszawa 1982.

[5] Instrukcja obsługi T-11 Tester typu tarcza-trzpień, Instytut Technologii Eksploatacji, Państwowy Instytut Badawczy, Radom 2008.

[6] K. Niewiarowski, Tłokowe silniki spalinowe, WKiŁ, **345-346**, Warszawa 1968.

[7] M. Hebda, Trybologia, WKiŁ, **144**, Warszawa 1980.

[8] C.M. Taylor, Engine Tribology, Tribology series 26, Elsevier, **213**, Amsterdam 1993.

[9] M. Wieczorowski, Wykorzystanie analizy topograficznej w pomiarach nierówności powierzchni, Wydawnictwo Politechniki Poznańskiej, **429**, 2009.

[10] J. Petzing, J. Coupland, R.Leach, A national measurement good practice guide no.116, NPL Middlesex 2010.

[11] http://www.hartowniats.pl.

[12] http://www.bohler.pl.

[13] http://www.uddeholm.com.

[14] P. Andersson, J. Tamminenm, C-K. Sandström, Piston ring tribology. A literature survey. VTT Tiedotteita – Research Notes 2178. **82**, Espoo 2002.