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MOS_2/WS_2/FINELPN COMPOSITE LAYERS – A NEW APPROACH TO LOW FRICTIONAL COATINGS FOR PISTON RINGS

WARSTWY KOMPOZYTOWE MOS_2/WS_2/FINELPN – NOWE PODEJŚCIE DO POWŁOK O NISKIM TARCIU NA PIERŚCIENIE TŁOKOWE

Key words: piston rings, nitriding, low friction, wear.

Abstract: In the search for alternative processes of technologically difficult and environmentally dangerous galvanic chromium coatings on cast iron piston rings, composite coatings of low-friction nanoparticles MoS_2, WS_2 and/or rGO embedded in a hard matrix of iron nitrides have been investigated. Laboratory tribological and operational tests on a real aircraft engine have been carried out to select the optimal technological parameters and microstructures of low frictional layers. A dry friction coefficient of 0.13 has been obtained for the best of the processes, i.e., four times lower than for raw reference samples (0.55). According to that technological option, the low-friction layers were produced for a set of rings for three cylinders of a Boxer aircraft engine and tested comparatively in an operational test with the rings in the other three cylinders. The experimental engine has passed the operational test obtaining the assumed power performance, fuel consumption and admission criteria of exhaust purity and oil purity.

Słowa kluczowe: pierścienie tłokowe, azotowanie, warstwy niskotarciowe, zużycie.

Streszczenie: W poszukiwaniu alternatywnych procesów dla trudnych technologicznie i niebezpiecznych dla środowiska galwanicznych powłok chromowych na żeliwnych pierścieniach tłokowych badano powłoki kompozytowe z niskotarciowych nanocząstek MoS_2, WS_2 i/lub rGO osadzonych w twardej osnowie z azotków żelaza. Przeprowadzono laboratoryjne badania tribologiczne oraz próby eksploatacyjne na rzeczywistym silniku lotniczym w celu doboru optymalnych parametrów technologicznych i mikrostruktur warstw o niskim współczynniku tarcia. Dla najlepszego z procesów uzyskano współczynnik tarcia suchego na poziomie 0,13, czyli czterokrotnie niższy niż dla surowych próbek referencyjnych (0,55). Zgodnie z tą opcją technologiczną wytworzono warstwy o niskim współczynniku tarcia dla zespołu pierścieni do trzech cylindrów silnika lotniczego Boxer i przetestowano je porównawczo w próbie eksploatacyjnej z pierścieniami chromowanymi w pozostałych trzech cylindrach, uzyskując założone parametry mocy, zużycie paliwa oraz kryteria dopuszczenia czystości spalin i oleju.

INTRODUCTION

The level of fuel consumption has a significant impact on the economy of piston engines in small airframes. It is well known that the reduction in efficiency in a piston engine is due to the friction between the piston and cylinder liner. The level of friction also affects the durability and operating parameters of the engine and determines the periods between repairs. Piston rings also transfer heat from the piston to the cylinder and constitute

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a significant seal of the combustion chamber **[L. 1, 2]**. Therefore, the piston-liner systems' low frictional and reliable behaviour is a significant technological issue. The new challenge aims at decreasing the friction coefficient between them even twice.

Intricate working conditions of piston rings **[L. 1–4]** require the use of advanced engineering materials **[L. 5]** and surface treatment **[L. 6, 7]**. The most commonly used engineering material for producing piston rings is nodular iron, which is usually electroplated with chrome to protect against scuffing and wear. However, due to the presence of Cr6+ ions **[L. 8]**, the electroplating technologies are the most dangerous for the environment and staff. Therefore, alternative concepts of protective coatings, such as flame **[L. 9]** or plasma **[L. 10–14]** spraying and various PVD or CVD coatings **[L. 15–17]**, are still being considered. However, none of the above-described alternative technologies to chromium plating have so far found widespread industrial application for the coating of cast iron piston rings. Alternatively, the gas nitriding process is also used for the surface hardening of rings, mainly steel ones **[L. 17, 18]**. This process significantly improves a wide range of tribological properties of the ring surfaces, reducing, inter alia, the friction coefficient and increasing the resistance to hydrogen wear **[L. 19–24]**. It is much more challenging to develop cast iron nitriding technology due to the presence of graphite in the material's structure **[L. 25, 26]**. An alternative to this application can be low-pressure nitriding (FineLPN), carried out in "boost-diffusion" cycles. This non-equilibrium process, both when applied to steel and cast iron, is supported by a dedicated computer application based on a neural network **[L. 27]** and numerous numerical simulations **[L. 28, 29]**. The list of potential thermochemical technologies for forming surface layers with a low friction coefficient is completed by the gas-sulphur nitriding technology widely used in the automotive and machine industries **[L. 30, 31]**.

A new concept of hybrid protective coatings for piston rings has been recently developed and presented **[L. 32, 33]**. It is based on the multistage process for creating a structurally optimised nitriding layer that is additionally enriched with inclusions of low frictional micro particles like MoS_2, WS_2 and rGO in the external nitrides zone. The preliminary research determined the background and basic parameters of new hybrid technology and revealed promising tribological behaviour of MoS_2/WS_2/FineLPN coatings on the S14 standard ductile cast iron **[L. 33]**. The present paper, as the first, describes the results of comparative tribological tests for different variants of this new technology made to optimise the microstructure and low frictional properties of a hybrid layer. The paper also describes the results of comparative engine tests, which were performed to compare the operating performance of piston rings coated by new hybrid surface layers versus traditional Cr galvanic coatings.

EXPERIMENTAL METHODS

Material and specimens

Cylindrical samples with dimensions of ø 24 mm x 8 mm were used in tribological tests, while industrial piston rings (ø 117.5 mm x 2.68 mm x 4.60 mm) were used in engine operation tests. The substrate material for all the specimens and piston rings used for the research was the industrially manufactured S14 grade standard ductile cast iron, according to the ISO MC 53.

All specimens and piston rings were heat-treated first to obtain the proper matrix microstructure, namely tempered martensite without any carbides' precipitations. Afterwards, all specimens for microstructural characterisation and tribological tests were divided into seven groups. The structural and technological status of these groups is summarised in **Table 1**. Low-friction layers were produced on samples from groups 4–7 according to the technical diagram presented in **Fig. 1** and details described in **[L. 32, 33]** and **Table 1**, using various combinations of low-friction reinforcing particles and thermochemical processes. Groups 1-3 contained reference samples from the basic

Table 1. Status of specimens for tribological tests
Tabela 1. Oznaczenie próbek do badań tribologicznych

Group no.	Thermochemical technology	Low frictional particles
1	Raw reference specimens	
2	LPN (12h)	–
3	AS (12h)	–
4	LPN (12h)	rGO
5	LPN (12h)	WS_2
6	LPN (12h)	MoS_2
7	LPN (8h) + AS (4h)	$MoS_2 + WS_2$

material – cast iron S14 (1), and from the same material after the component undergoing thermo-chemical processes of low-pressure nitriding LPN (2) as well as gas sulphonitriding AS (3).

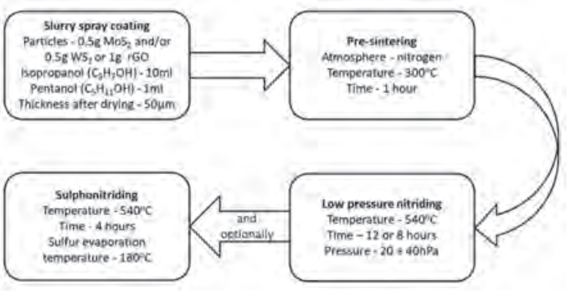

Fig. 1. Diagram of technology for producing low-friction layers

Rys. 1. Schemat technologii wytwarzania warstw niskotarciowych

It was assumed that low-friction layers would be produced on piston rings for in-service testing according to the best concept selected after tribological tests.

Microstructural characterisation

The metallographic investigations were carried out using the Nikon MA200 optical microscope. The microstructures in the cross-sections of the specimens were observed and registered before they were transferred for tribological tests. The distribution of chemical elements in the individual zones of the created hybrid layers was tested using the SEM JEOL JSM-6610 and the integrated EDS X-MAX Oxford Instruments spectrometer.

A cross-section microstructure of investigated low frictional layer is presented in **Figure 2**, an example of the specimen from group no. 7. The following sublayers have been presented sequentially from the surface:

- epitaxially grown iron nitride zone ε that contains built-in low friction inclusions MoS_2 and/or WS_2 – (3.5 µm),
- iron nitride zone ε + γ'(33.2 µm), with fine FeS inclusions near to the surface (9.7 µm),
- relatively deep (174 µm) dark zone that is reinforced by precipitation hardening.

In addition, the original inclusions of spheroidal graphite are built into a continuous layer of iron nitrides, creating additional dry lubricant micro storage tanks. The loosening of the surface structure is visible in the SEM image, while EDS analysis reveals the increased concentration of elements constituting components of low-friction inclusions (Mo, W, S) in the hard matrix of iron nitrides up to a depth of approx. 3 mm (**Fig. 3**).

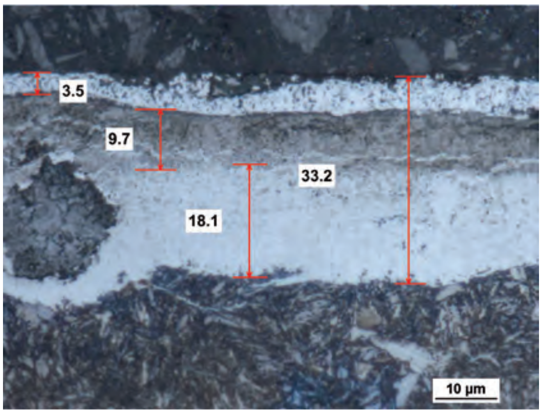

Fig. 2. Microstructure of low frictional layer on the specimen from group no. 7

Rys. 2. Mikrostruktura warstwy niskotarciowej próbki z grupy 7

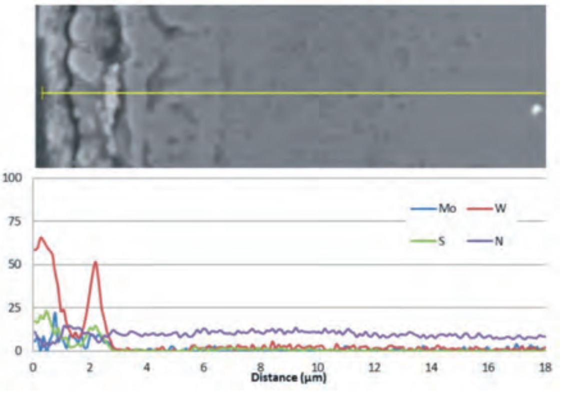

Fig. 3. SEM picture and EDS concentration plots for Mo, W, S and N in the surface layer

Rys. 3. Obraz SEM i wykresy stężenia EDS dla Mo, W, S i N w warstwie powierzchniowej

The structure of the multi-zone surface layer that is suited in this way should be appropriate for lowering the coefficient of friction and increasing the wear resistance of the piston rings made of ductile iron.

Tribological tests

Tribological tests with oscillatory motion were carried out in dry friction conditions with the contact geometry "ball on disc". A tribometer SRV Optimal (Instruments Prüftechnik) was applied. The choice of the research method was dictated by the similarity of the friction nature to the real object, i.e. in the piston positions with reciprocating motion. The following experimental conditions were taken:

- normal load – 20 N,
- test cycle stroke – 1 mm,
- frequency – 20 Hz,
- 1800s total test time for each sample,
- temperature – 25°C.

During the test, the dry friction coefficient was recorded continuously. After completing the test, the maximum depth of the friction marks in the tested layer was measured. SEM also observed the friction mark morphology. The measurement series for each tested option constituted three samples, and the counter-sample was a 100Cr6 φ10 mm hardened steel ball. The average values of the friction coefficient were determined from the friction diagrams for each tested variant (total test duration – 5400 s for all tested samples). The mean values of the maximum depths of the friction paths were measured, and their standard deviations were calculated alike.

In-service engine tests

The in-service engine tests were carried out in the stand for operational tests, based on the Franklin 6A-350-C1 piston engine – six-cylinder, 4-stroke, clockwise rotation, air-cooled, in the "boxer" system, intended for use as light aircraft propulsion (**Fig. 4**). Operating cycle conditions were of 200 hours long trial according to the following program:

- Stage 1 – 5 h, slow speed 600–800 RPM;
- Stage 2 – 5h , max revolutions, max torque;
- Stage 3 – 5 h, max speed, 75% torque;
- Stage 4 – 150 h – rated speed 2500 – 2700 RPM, torque 75%;
- Stage 5 – 35 h, rated speed 2500 – 2700 RPM, max torque.

Fig. 4. View of the 6A-350-C1 engine used in operational tests
Rys. 4. Widok silnika 6A-350-C1 używanego w testach eksploatacyjnych

Two types of piston rings were tested simultaneously, namely those with hybrid low-friction layers produced according to the parameters of research group no. 7 (cylinders no. 1, 2, 3) and those with reference to standard chrome coatings (cylinders no. 4, 5, 6). During the whole test, fuel consumption and oil consumption were determined for the entire engine, and exhaust gas was tested for compliance with EASA Annex 16, vol. 2, 07/2008, as well as oil pollution for compliance with ASTM D6595 – 16. Each cylinder recorded the oil, head, and exhaust gas temperature separately. After the test, the engine was dismounted, and cylinder wear measurements were carried out on the measuring diameters, as shown in **Fig. 5**. The measurements were performed on each of the diameters A, B and C in two perpendicular planes. A total of 6 measurements were made for each cylinder.

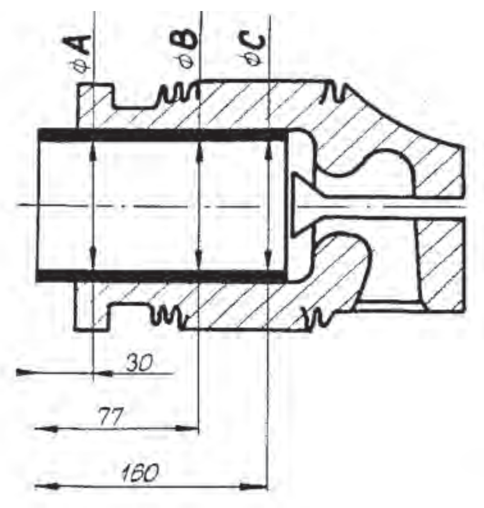

Fig. 5. Location of operational wear measurements
Rys. 5. Miejsca pomiarów zużycia eksploatacyjnego

EXPERIMENTAL RESULTS

Tribological tests

Examples of friction coefficient plots recorded during tribological tests are presented in **Fig. 6**. Each of the graphs for samples with a modified surface structure (groups no. 2–7) was compared with a friction graph for the raw reference sample (no. 1). The values of the average coefficient of friction (a.f.c.) determined for each three-sample series are also given on each graph. It is easy to see that all the modifications of the ductile iron used resulted in a significant decrease in the dry friction coefficient. The friction graphs for these samples are definitely smoother in relation to the reference sample, which indicates a significant reduction of seizing in micro-areas. A strong low friction effect was first observed by introducing alternatively lubricating nanoparticles with a layered structure (rGO, MoS_2 or WS_2) into the iron nitride matrix during the LPN low-pressure nitriding process. An effect on a similar scale was achieved using a gas sulfonitriding process in which one simultaneously saturates the iron surface with sulphur and nitrogen, forming a structure of iron nitrides with inclusions of FeS hexagonal iron nitrides **[L. 30, 31]**. However, the best tribological properties were obtained by using synergistic reinforcement with MoS_2 and WS_2 particles, which were embedded in the nitride matrix in the sequence of low-pressure nitriding and gas sulphonitriding processes (group no. 7). The average coefficient of friction for this group (0.13) is lower by half than for samples reinforced with a single low-friction phase (0.23–0.28) and four times lower than the one obtained for raw reference samples (0.55).

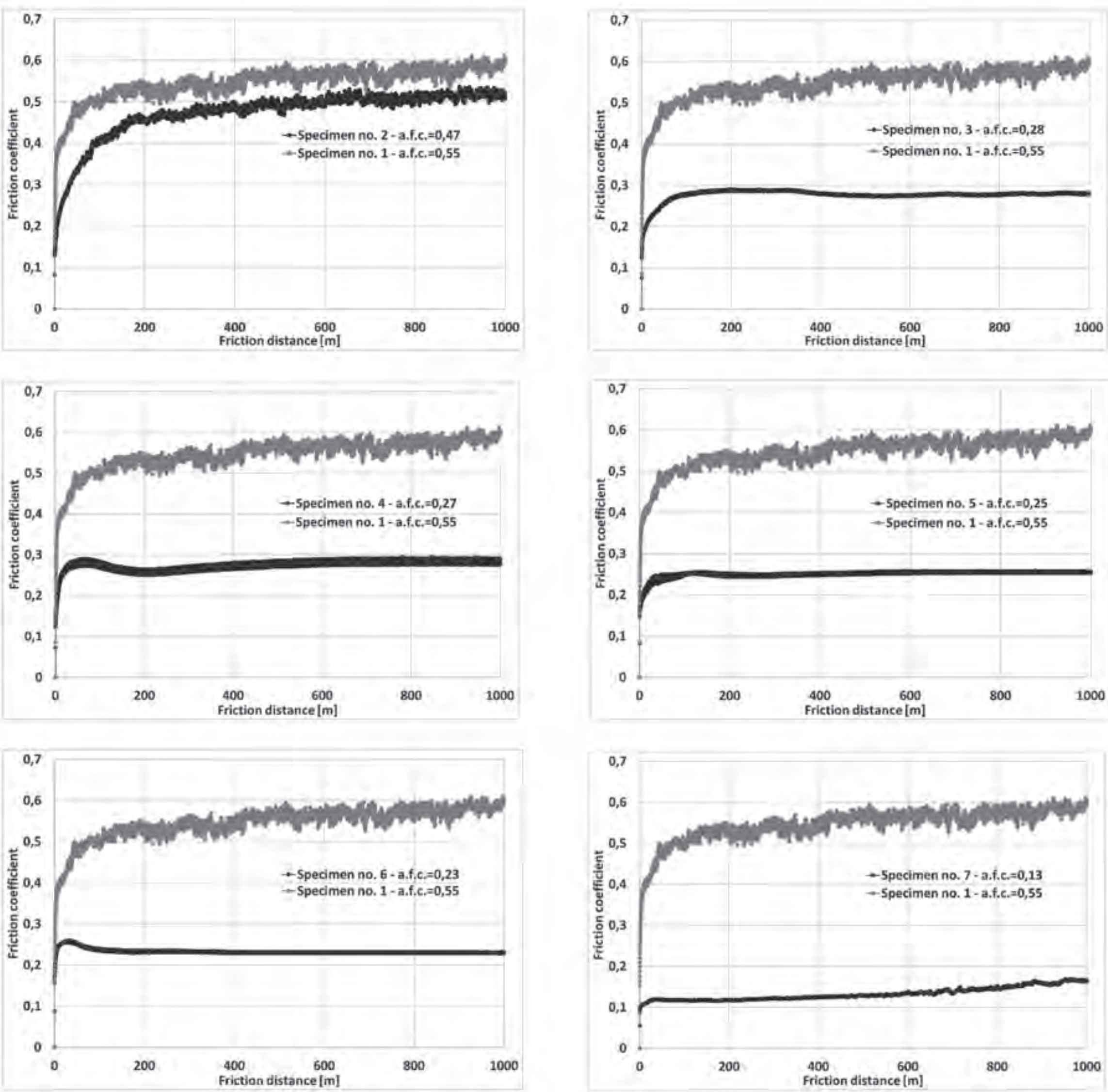

Fig. 6. Friction coefficient plots recorded during tribological tests
Rys. 6. Wykresy współczynnika tarcia zarejestrowane podczas testów tribologicznych

The images of friction marks (**Fig. 7**) and their depth (**Fig. 8**) correlate well with the measured values of the friction coefficient. Structures without the inclusion of low-friction particles (Samples No. 1 and No. 2) show deep grooves resulting from adhesive affinity and consequent scuffing. Structures containing at least one low-friction phase (samples 3–6) show less tendency to scuffing.

As a consequence, the friction marks are much smoother and shallower. A valuable added value to low-friction properties is the synergistic interaction of three types of nitride matrix inclusions, namely MoS_2, WS_2 and FeS (sample No. 7), which results in an absolutely smooth and extremely shallow trace of friction after the tribological test.

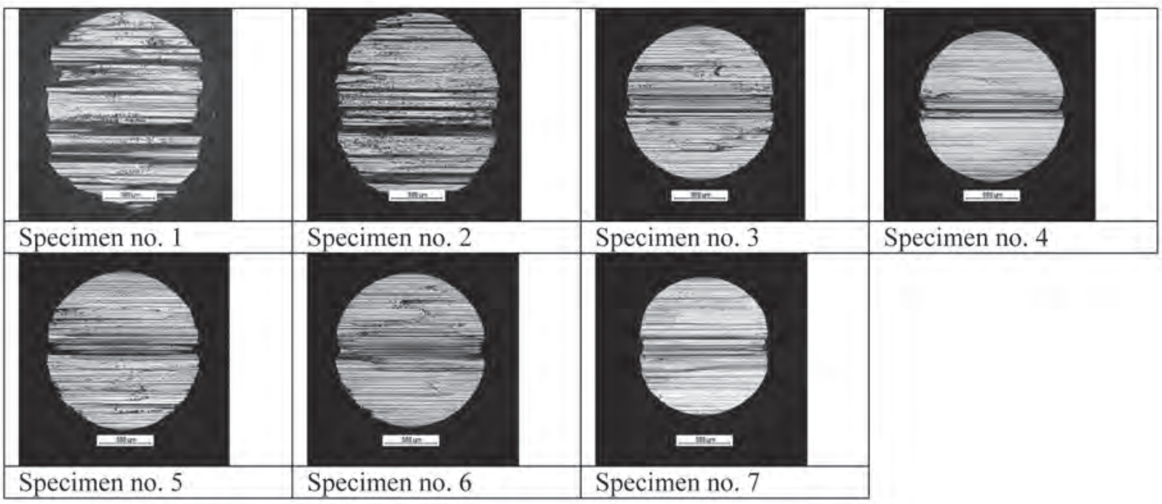

Fig. 7. Macroscopic view of friction marks representative of individual measurement groups
Rys. 7. Widok makroskopowy śladów tarcia reprezentatywnych dla poszczególnych grup badawczych

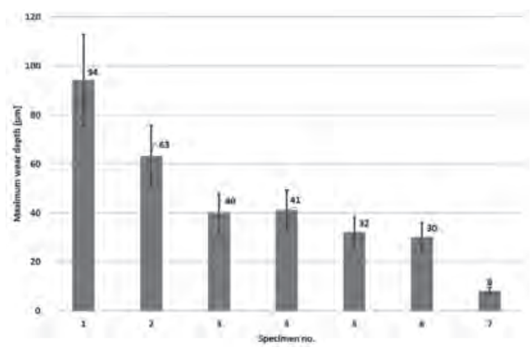

Fig. 8. Results of the depth of friction marks measurements after tribological tests
Rys. 8. Wyniki pomiarów głębokości śladów tarcia po badaniach tribologicznych

As a result of laboratory tribological tests, it has been found that samples demonstrated the best low-friction and anti-wear properties from measuring group No. 7. Therefore, according to this technological plan, piston rings were processed for comparative operational tests on a real engine.

In-service engine tests

During the engine test, fuel consumption and oil consumptions were 0.375 g/ kWh and 0.26 kg/h, respectively. These values did not exclusively differ from those obtained for tests with standard chromium-coated rings. Also, the exhaust gas composition and the oil impurities met the EASA guidelines and ASTM standards, respectively. In all stages of the engine test, slightly lower oil temperature (**Fig. 9**) and temperature of the heads (**Fig. 10**) were recorded in cylinders with rings with low-friction layers compared to cylinders equipped with traditional chromium-plated rings. Although these differences are small (around 2°C), they are repeatable and systematic. They can be attributed to the lower friction between piston rings and a cylinder liner.

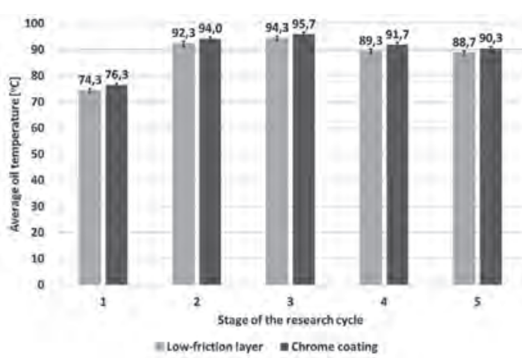

Fig. 9. Comparison of average oil temperature in cylinders between rings with low friction layers and rings with chromed layers in subsequent stages of the operational test
Rys. 9. Porównanie średniej temperatury oleju w cylindrach pomiędzy pierścieniami z warstwami o niskim współczynniku tarcia i pierścieniami z warstwami chromowanymi w kolejnych etapach próby eksploatacyjnej

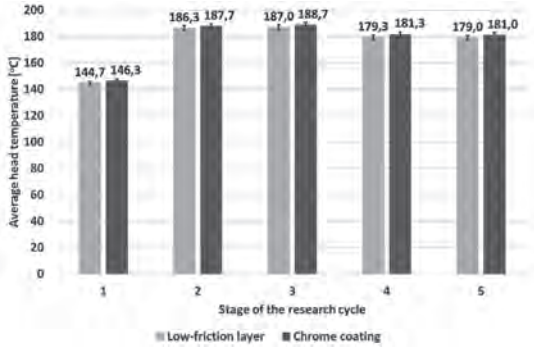

Fig. 10. Comparison of average cylinder head temperature in cylinders between rings with low friction layers and rings with chromed layers in subsequent stages of in service testing

Rys. 10. Porównanie średniej temperatury głowic w cylindrach pomiędzy pierścieniami z warstwami o niskim współczynniku tarcia i pierścieniami z powłokami chromowanymi w kolejnych etapach badań eksploatacyjnych

After dismounting the tested engine, it was revealed that the friction trace in cylinders cooperating with low friction layers was slightly shallower compared to reference cylinders along the entire length of the piston stroke (**Fig. 11**).

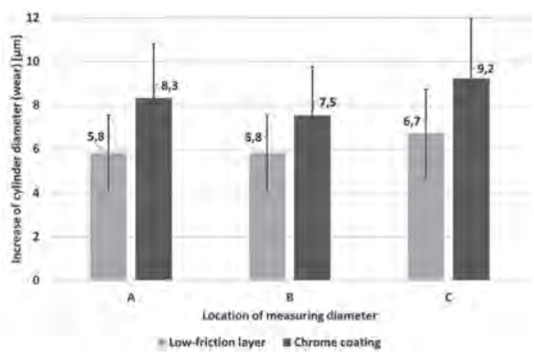

Fig. 11. Comparison of the wear depth of the cylinder liners at three points along the piston stroke length

Rys. 11. Porównanie głębokości zużycia tulei cylindrowych w trzech punktach na długości skoku tłoka

DISCUSSION

As a result of tribological studies, it has been found that the necessary condition for obtaining anti-seizing, low-friction properties of the iron nitride zone on ductile iron is the incorporation of nano/mezo inclusions with a layered structure into their structure. The absence of layered inclusions in samples exclusively low-pressure nitriding (group no. 2) is the reason for only a slight decrease in the coefficient (0.47) compared to the raw reference samples (0.55). The incorporation of only one low-phase phase in the nitrides, i.e. FeS (group No. 3), rGO (group No. 4), WS_2 (group No. 5), and MoS_2 (group No. 6) results in a repetitive, approximately double reduction of the friction coefficient, 0.28, 0.27, 0.25 and 0.23, respectively. The lowest friction coefficient was obtained by double modification with MoS_2 and WS_2 particles, which were embedded in the nitride matrix in the sequence of low-pressure nitriding and gas sulphonitriding processes (0.13). Probably the gas sulphonitriding process in the final phase of the heat-chemical treatment cycle introduces significant added value to the low-friction properties. Firstly, it introduces into the structure of hard nitrides a third type of low-friction inclusions with a layered structure, namely hexagonal iron nitride FeS **[L. 30, 31]**. Secondly, the gas sulphonitriding process causes a significant loosening of the surface structure while maintaining a hard nitride matrix **[L. 30, 31]**. Thirdly, the annealing of the composite structure with WS_2 and MoS_2 inclusions during the final stage of the technological process in the presence of sulphur vapours can improve their layer structure by crystallisation of local amorphous regions **[L. 30, 31]**. All the mechanisms listed above should result in a decrease in adhesive affinity and a reduction in shear strength while maintaining a high yield point of the surface layer, which according to Bowden's theory **[L. 34]**, will significantly reduce the coefficient of friction. However, the hypothesis presented above requires confirmation of high-resolution nano and micro-structural research; the low-friction effect obtained is spectacular and repeatable. Its suitability for surface treatment of aircraft piston rings has been confirmed in the framework of in-service engine tests. Thus, the process of manufacturing hybrid low-friction layers developed and technologically optimised as part of the presented research may be an alternative to galvanic technology of chromium plating of piston rings and other elements working in friction conditions.

CONCLUSIONS

1. Low friction gradient layers which contain nanoinclusions of MoS_2 and/or WS_2 solid lubricants in nitride matrix can be a prospective alternative for chromium-based galvanic coatings on piston rings made of ductile cast

iron due to the elimination of toxic ions Cr+6 from production.
2. The best low frictional and anti-wear properties were obtained using synergistic reinforcement with MoS_2 and WS_2 particles, which were embedded in the nitride matrix in the low-pressure nitriding and gas sequence sulphonitriding processes.
3. The engine equipped with half rings with a low friction layer has passed the operational test obtaining the assumed power performance, fuel consumption and admission criteria of exhaust purity and oil purity.
4. A slightly lower oil temperature and head temperature were found in the cylinders equipped with rings with a friction layer, which was probably the result of a lower coefficient of friction at the interface between the piston ring and cylinder liner.
5. The friction traces in cylinders coworking with low friction layers were slightly shallower compared to reference cylinders.
6. The structure of lubricating nanoparticles embedded in a hard matrix of iron nitrides, diffusively bonded to the substrate by a gradient interface, is the key to low friction properties and durability of the coatings.

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