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INDUSTRIAL SCALING OF LOW-FRICTION HYBRID LAYER TECHNOLOGY ON PISTON RINGS

PRZEMYSŁOWE SKALOWANIE TECHNOLOGII NISKOTARCIOWYCH WARSTW HYBRYDOWYCH NA PIERŚCIENIACH TŁOKOWYCH

Key words: piston rings, nitriding, low-friction layers, distortions.

Abstract: Technological trials of gas sulphonitriding processes for cast iron piston rings were carried out as an alternative to chromium electroplating for the production of low-friction layers. The effectiveness of different ways of packing heat treatment charges in minimising or eliminating deformation of the rings was compared, and the effectiveness of ring packing with restraining tooling was confirmed. The maximum duration of the gas sulphonitriding process was determined at 1 hour, both as a stand-alone process and as a component process in a multi-stage technology for the manufacture of low-friction hybrid layers, ensuring the maintenance of sufficient ring toughness for an opening during assembly.

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

Streszczenie: Przeprowadzono próby technologiczne procesów azotonasiarczania gazowego żeliwnych pierścieni tłokowych jako alternatywy wytwarzania warstw niskotarciowych dla chromowych powłok galwanicznych. Porównano skuteczność różnych sposobów upakowania wsadów do obróbki cieplno-chemicznej w minimalizacji bądź eliminowaniu odkształceń pierścieni. W tym zakresie potwierdzono skuteczność pakietowania pierścieni w specjalistycznym, krępującym oprzyrządowaniu technologicznym. Wyznaczono maksymalny czas trwania procesu azotonasiarczania gazowego – 1 godziny – zarówno jako procesu samodzielnego, jak i procesu składowego w wieloetapowej technologii wytwarzania niskotarciowych warstw hybrydowych, który gwarantuje zachowanie dostatecznej wiązkości pierścienia dla rozwarcia montażowego**.**

INTRODUCTION

In the search for alternatives to chromium plating for the production of low-friction layers on aircraft engine piston rings, simple and hybrid processes using diffusive saturation of surface layers with nitrogen are increasingly being considered and implemented. The gas nitriding process is already widely used for surface hardening rings, mainly steel-made **[L. 1–3]**. However, a considerable technological challenge is applying thermo-chemical treatment to cast iron rings due to their structural multiphase nature and, above all, the occurrence of graphite inclusions in the microstructure **[L. 4–6]**. A new solution in this area is the concept of producing hybrid low-friction layers on aircraft piston rings **[L. 7]**. This entails the formation of a structurally optimised nitrided

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layer that contains, in addition, inclusions of low-friction microparticles such as MoS_2, WS_2 and rGO, which are embedded in the outer zone of iron nitrides. Preliminary studies showed promising tribological properties of MoS_2/WS_2/FineLPN coatings on ductile iron of S14 grade **[L. 8]**. In the next stage of development of this technological concept, comparative tribological studies were carried out to select optimal microstructures associated with low-friction properties of the surface layers. For the friction-optimal structures of the hybrid layers, comparative engine field tests of the rings were carried out. They revealed the performance parameters of rings with MoS_2/WS_2/FineLPN coatings as complementary to traditional chromium coated rings **[L. 9]**.

A necessary condition for large-scale scaling of the technology is the development of detailed technological documentation, including the ranges of permissible technological parameters and optimisation of the packing of ring charges, including the use of specialised auxiliary equipment. A key issue is the elimination of post-process deformation and the achievement of sufficient material toughness to enable ring assembly and, above all, the repeatability of manufacturing results. These research and development issues were addressed in this study through the performance and interpretation, as a simplification, of a series of experimental gas sulphonitriding processes **[L. 10, 11]** of aerospace piston rings made of S14 grade ductile iron.

EXPERIMENTAL METHODS

Materials, specimens and processes

Industrially manufactured piston rings (trapezoidal and oil rings), made of ductile cast iron of S14 grade according to ISO MC 53, were subjected to three variant gas sulphonitriding processes, carried out using the production equipment at Hart-Tech Co. Ltd. Basic parameters of these processes are summarised in **Table 1**.

Table 1. Basic parameters of sulphonitriding processes
Tabela 1. Podstawowe parametry procesów azotonasiarczania gazowego

Process	Temperature	Time
I	540°C	8 h
II	540°C	2 h
III	540°C	1 h

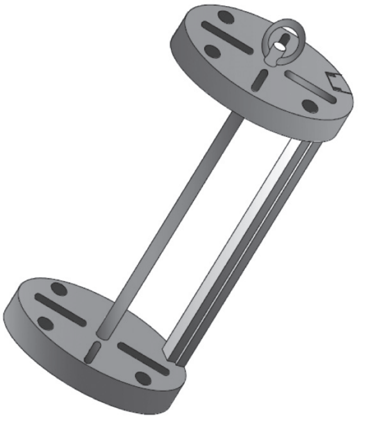

Fig. 1. Concept of specialised tooling for the stacking of rings
Rys. 1. Schemat specjalistycznego oprzyrządowania do pakietowania pierścieni

In Process I, the rings were treated using four tested options of stacking in the retort:
- Option A – No bundling – rings laid out loosely, individually on the grid;
- Option B – Rings stacked but not top-loaded;
- Option C – Rings stacked and top-loaded;
- Option D – Rings stacked and pressed from above according to the patent **[L. 12]**.

A diagram of the patented process tooling used for rings treated in Option D is shown in **Fig. 1**. The tooling includes two creping circular pressure plates, and the pressure in the ring pack is controlled by an axial compression screw. A system of holes in the pressure plates allows the process atmosphere to circulate inside the ring pack during the treatment. This enables the formation of a sulphonitrided layer on the inner surface of the rings, which provides a stress compensation area for the low-friction surface layer. This should significantly reduce the post-process deformation of the sulphonitrided piston rings.

For all variants of ring stacking in process I, measurements of dimensions and deformations were made in order to select the optimum technological tooling option. The values were measured and compared statistically, and on this basis, it was found that a technologically satisfactory level of deformation was obtained only for the batch of rings packaged in accordance with option D. For none of the groups of rings subjected to process I was sufficient toughness obtained to enable the rings to be assembled in the piston grooves. Hence, in the subsequent, significantly shortened sulphonitriding processes (processes II and III), the rings were bundled for treatment only using the specialised tooling (Option D). For rings treated in process II, satisfactory bonding was also not obtained; therefore, this group of rings was not subjected to detailed deformation tests. Sufficiently high material toughness was demonstrated for the group of rings machined in the shortest process (III). This group, numbering 30, underwent detailed geometry measurements.

EXPERIMENTAL RESULTS

Structural characterisation

Metallographic investigations of the treated piston rings were carried out using a Nikon MA200 optical microscope.

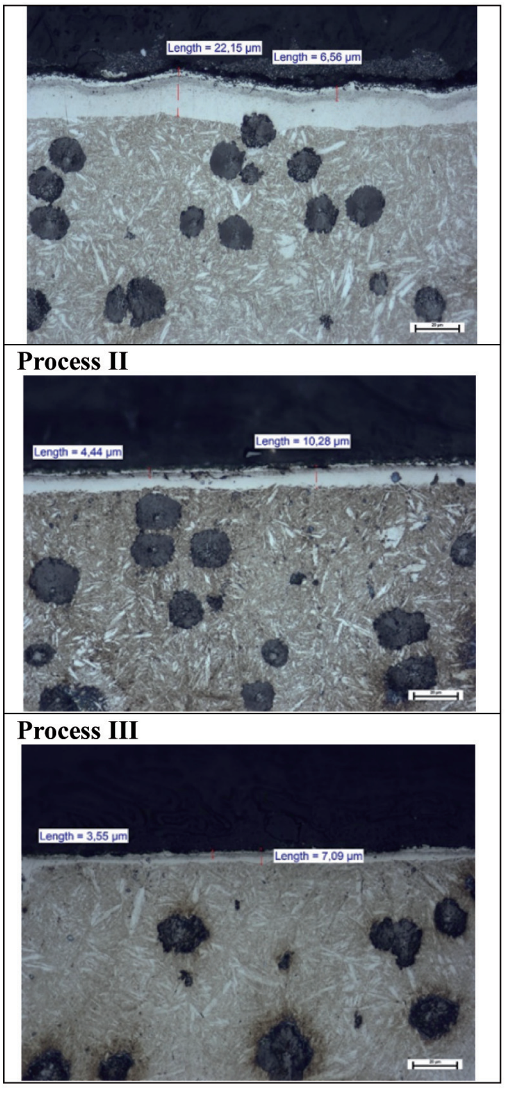

Fig. 2. Microstructures of low-friction surface layers on piston rings created as a result of processes I, II and III

Rys. 2. Mikrostruktury niskotarciowych warstw powierzchniowych na pierścieniach tłokowych, wytworzone w procesach I, II i III

The microstructures in the cross-sections of the piston rings were observed and registered. They are presented in **Fig. 2**. Microhardness HV01 distributions in the surface layers were also determined in the cross-sections of the treated piston rings using a KB PRÜFTECHNIK microhardness tester.

The microhardness plots obtained are presented in **Fig. 3**. The effective case depth of surface hardening was determined according to the criterion of 150HV above the core hardness. The estimated hardening case depths are illustrated in **Fig. 4**.

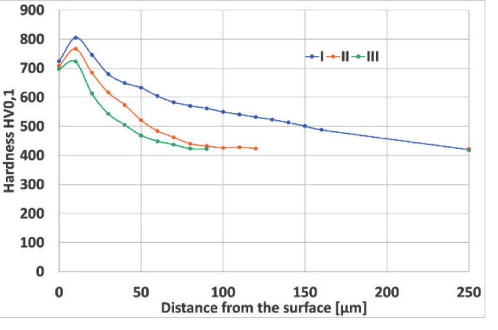

Fig. 3. Microhardness plots of low-friction layers obtained as a result of processes I, II and III

Rys. 3. Rozkłady mikrotwardości warstw niskotarciowych wytworzonych w procesach I, II i III

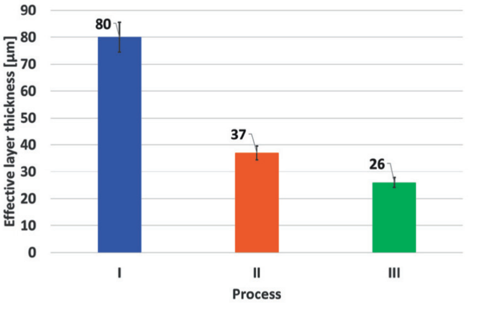

Fig. 4. Effective case depth of low-friction layers obtained as a result of processes I, II and III

Rys. 4. Efektywna grubość warstw niskotarciowych wytworzonych w procesach I, II i III

Measurements of dimensions and distortions

The sulphonitrided trapezoidal rings were geometrically measured to determine the following:

– the lock gap;
– tangential force, i.e., the force exerted by the ends of the ring lock when closing the ring in the steel band;
– circularity;
– circumferential light tightness.

The specialised measuring equipment for determining ring circularity and circumferential light tightness is shown in **Figures 5** and **6**, respectively.

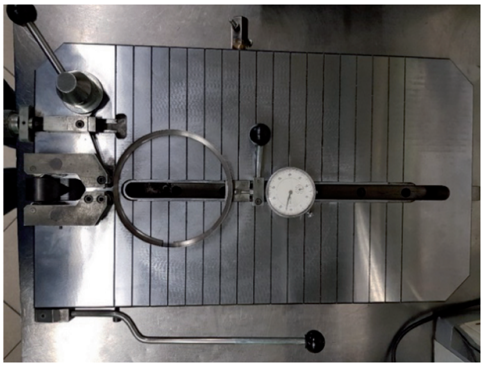

Fig. 5. Measuring stand for ring circularity
Rys. 5. Stanowisko do pomiaru owalizacji pierścieni

Fig. 6. Measuring stand for rings' circumferential light tightness
Rys. 6. Stanowisko do pomiaru przylegalności pierścieni

The stand shown in **Fig. 5**, equipped with a table, a dial indicator and steel tape, is used for circular measurements. Calibration consists in setting the length of the steel band that wraps around the ring. The length of the tape should be selected in such a way that after tightening it and closing the ring in it, the ring has the slot of the lock in accordance with the construction slot. Then the tip of the sensor approaches the track at a point 90^{o} from the slot of the lock; the sensor is then zeroed. Then the tape is loosened, and the ring is rotated so that after tightening the steel tape again, the slot of the ring lock is in the same axis as the tip of the dial indicator. We read the change in the position of the sensor pointer relative to the base position, which is also information about the circularity value.

The stand shown in **Figure 6** is used for the circumferential light tightness measurements of the rings. The ring is placed in a gauge with a dimension corresponding to the diameter of the internal combustion engine cylinder. The gauge with the ring is placed on the device consisting of an inclined table and a light source. The measurement consists in setting the gauge with the ring in such a way that it is possible to observe clearances (if any) between the inner race of the gauge and the outer race of the ring. The percentage of rings' circumferential light tightness is evaluated.

The oil rings were tested for the lock gap only. The results of measurements for the rings treated by process I are shown as graphs in **Figs. 7–11**.

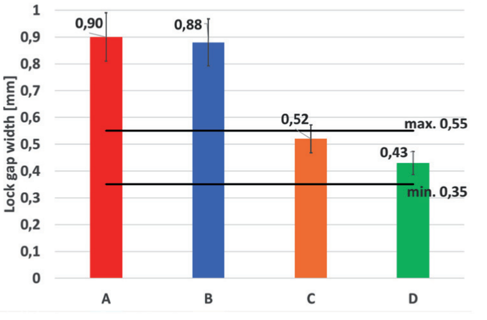

Fig. 7. Results of lock gap measurements of trapezoidal rings treated by process I
Rys. 7. Wyniki pomiarów szczeliny zamka pierścieni trapezowych obrobionych w procesie I

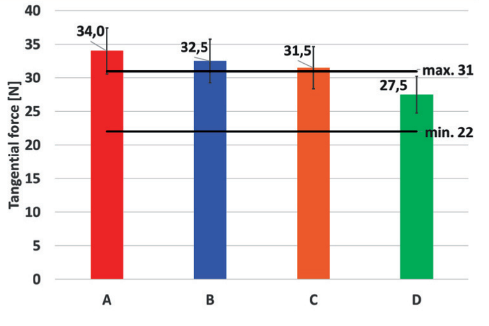

Fig. 8. Results of tangential force measurements of trapezoidal rings treated by process I
Rys. 8. Wyniki pomiarów siły stycznej pierścieni trapezowych obrobionych w procesie I

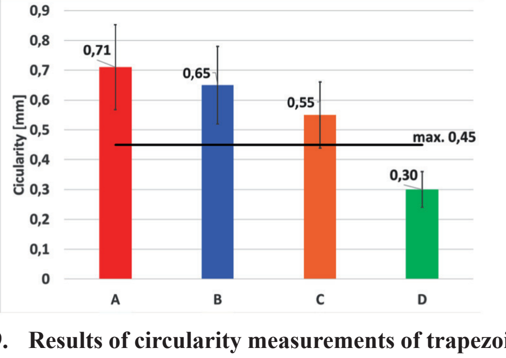

Fig. 9. Results of circularity measurements of trapezoidal rings treated by process I
Rys. 9. Wyniki pomiarów owalizacji pierścieni trapezowych obrobionych w procesie I

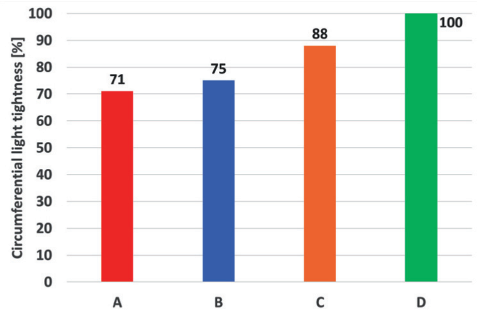

Fig. 10. Results of circumferential light tightness measurements of trapezoidal rings treated by process I

Rys. 10. Wyniki pomiarów przylegalności pierścieni trapezowych obrobionych w procesie I

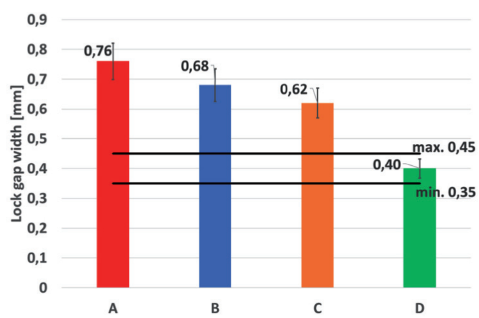

Fig. 11. Results of lock gap measurements of oil rings treated by process I

Rys. 11. Wyniki pomiarów szczeliny zamka pierścieni olejowych obrobionych w procesie I

The permissible ranges of values are marked in the above graphs according to **[L. 13–17]**. Designations A, B, C and D are described above.

The results of measurements for the rings treated by process III are given in **Tables 2** and **3**. After processes I and II, the rings did not meet the criterion of sufficient elasticity for the application in real friction pairs.

Table 2. Results of measurements for rings treated by process III

Tabela 2. Wyniki pomiarów pierścieni trapezowych po procesie III

Parameter	**Measurement results**	**Permissible range**
Lock gap [mm]	0.41 ± 0.02	0.35–0.55
Tangential force [N]	26.3 ± 0.5	22.0–31.0
Circularity [mm]	0.31 ± 0.07	0.00–0.45
Circumferential light tightness [%]	100 ± 0	min. 100

Table 3. Results of measurements for rings treated by process III

Tabela 3. Wyniki pomiarów pierścieni olejowych po procesie III

Parameter	**Measurement results**	**Permissible range**
Lock gap [mm]	0.39 ± 0.02	0.35–0.45

All deformation measurements were performed using 30 samples. The mean values and standard deviations are presented in the graphs and tables.

DISCUSSION

The scientific assumption for the research carried out was to achieve the objective of minimising post-process deformation of cast iron piston rings through the synergistic effect of restraining their movement during the thermo-chemical process and the symmetrical compensation of the residual structural stresses of the low-friction layer by the layer produced on the inner surface. The first attempt at industrial upscaling of the technology for producing sulphonitrided layers on piston rings made of S14 ductile iron demonstrated the need for their stacking to limit the thermal and structural deformation of the rings during thermochemical treatment. As a result of process, I, only rings packed using specialised tooling (**Fig. 1**) retained geometry and elasticity within the acceptable ranges (**Figs. 7–11**). However, the long duration of process I formed a relatively thick, brittle zone of ε-iron nitrides in the surface layer (22.15 µm), in which low-friction iron sulphide inclusions were embedded to a depth of 6.56 µm (**Fig. 2**). Such an extensive, brittle compound zone lowered the toughness of the rings, causing them to fracture during simulated assembly. The nucleation site for these cracks was the brittle compensation layer on the inner surface of the rings, loaded during assembly with tensile stresses.

In the subsequent, shorter sulphonitriding processes, the thickness of the brittle nitride zone was significantly reduced: to 10.28 µm in the two-hour process (II) and to 7.09 µm in the one-hour process (III) (**Fig. 2**). It should be noted that the approximately threefold reduction in the thickness of the brittle ε-nitrides zone was accompanied by only an approximately twofold reduction in the thickness of the zone containing low-friction iron sulphide inclusions (3.55 µm). It should also be emphasised that, despite significant differences in the effective thickness of the hardened layers produced, which ranged from 26 to 80 µm (**Fig. 4**), the surface hardness is similar (700–730 HV), as is the maximum hardness (730–800 HV), which is at the same depth of about 10 µm for all of the layers examined (**Fig. 3**). Thus, short-duration gas sulphonitriding (1 h) resulted in a low-friction layer that was correctly formed in terms of tribological requirements, with a thickness of about 3.5 µm, ensuring good interoperation of the rings with the cylinder, while at the same time providing sufficient material toughness to allow the rings to be installed in the piston grooves. Testing a batch of 30 sulphonitrided rings for one hour in dedicated instrumentation confirmed that geometry and elasticity were maintained statistically within acceptable value ranges (**Tables 2** and **3**). This confirmed the validity of the postulated concept of synergistic interactions in the deformation limiting of cast iron piston rings during the sulphonitriding process. The controlled stacking of the rings eliminated the risk of thermal deformation of the rings during the process and protected the flat surfaces of the rings from reactant diffusion and structural changes. In this way, the potential stress component causing a loss of flatness of the rings was eliminated. The formation of a compensation layer on the inner surface minimised ring deformation to a level acceptable in the technical specification **[L. 13–17]**.

With the help of the tooling used, low-friction layers with a small thickness of about 3.5 µm, but sufficient from the point of view of acceptable wear of the rings **[L. 18]**, were obtained. Their competitive advantage over traditional chromium electroplating coatings is not only the excellent tribological properties studied previously **[L. 8]** but also the diffusive cohesion of the layer with the substrate, which excludes the risk of coating delamination during operation **[L. 9]**. It has been identified that the limitation of the maximum thickness of the sulphonitrided layer is due to the brittleness of the compensating layer. There is, therefore, the potential to enlarge the acceptable thickness of the external low-friction layer by a controlled limitation of the growth of the internal compensation layer. The method for achieving this is to further experimentally improve the process tooling towards a controlled reduction in the circulation of the processing atmosphere inside the ring pack.

CONCLUSIONS

1. The production of low-friction layers on cast iron piston rings in simple and hybrid processes incorporating thermo-chemical nitrogen saturation requires the use of dedicated restraint tooling.
2. Dedicated process instrumentation ensures that ring geometry and elasticity are maintained within an acceptable range by the synergistic effect of restraining their movement during the thermo-chemical process and the symmetrical compensation of the inherent structural stresses of the low-friction layer by the layer formed on the inner surface.
3. The processing time for gas sulphonitriding of piston rings made of ductile cast iron of S14 grade should be about 1 hour.
4. As a result of such a process, well-developed composite low-friction layers with a limited thickness of the brittle nitride zone are obtained, which guarantees the maintenance of sufficient material toughness to enable the rings' assembly.

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REFERENCES

1. Saravanan R., Sunkara N., Rao M.S., Malyadrib T.: Experimental exploration on influence of gas nitriding and chromium coated piston rings in reduction of wear and lubricant consumption in petrol engines. AIP Conference Proceedings, 2020, vol. 2283(1), p. 20071, doi.org/10.1063/5.0025022.
2. Michalski J., Wach P., Tacikowski J., Betiuk M., Burdynski K., Kowalski S., Nakonieczny A.: Contemporary industrial application of nitriding and its modifications. Materials and Manufacturing Processes, 2009, vol. 24(7–8), pp. 855–858, doi.org/10.1080/10426910902844203.
3. Xu B., Yin B., Gao D., Hua X.: Tribological performance of surface treated piston assembly with infiltrated layer. Journal of Mechanical Science and Technology, 2022, vol. 36(1), pp. 197–204, doi.org/10.1007/s12206-021-1218-4.
4. Wołowiec-Korecka E., Kula P., Pawęta S., Pietrasik R., Sawicki J., Rzepkowski A.: Neural computing for a low-frictional coatings manufacturing of aircraft engines' piston rings. Neural Computing and Applications, 2019, vol. 31, pp. 4891–4901, doi.org/10.1007/s00521-018-03987-9.
5. Watanabe S., Noshiro J., Miyake S.: Tribological characteristics of WS2/MoS2 solid lubricating multilayer films. Surface and Coatings Technology, 2004, vol. 18(2–3), pp. 347–351, doi.org/10.1016/j.surfcoat.2003.09.063.
6. Writzl V., Rovani A.C., Pintaude G., Lima M.S.F., Guesser W.L., Borges P.C.: Scratch resistances of compacted graphite iron with plasma nitriding, laser hardening, and duplex surface treatments. Tribology International, 2020, vol. 143, p. 106081, doi.org/10.1016/j.triboint.2019.106081.
7. Kula P., Pietrasik R., Paweta S.: Low-friction layer from nanocomposite gradient material and method for producing it. Patent Office of the Republic of Poland. Patent No. PL 412975, 2019.
8. Kula P., Pietrasik R., Pawęta S., Rzepkowski A.: Low Frictional MoS2/WS2/FineLPN Hybrid Layers on Nodular Iron. Coatings, 2020, vol. 10(3), p. 293, doi.org/10.3390/coatings10030293.
9. Kula P., Pietrasik R., Pawęta S., Komorowski J.: MoS_2/WS_2/FineLPN composite layers – a new approach to low frictional coatings for piston rings. QUARTERLY TRIBOLOGIA, 2022, 301(3), pp. 49–58, DOI: 10.5604/01.3001.0016.1024.
10. Has Z., Kula P., Gawronski Z.: Structural construction of sulfonitrided layers in stainless and heat-resisting steels. Archives of Materials Science, 1980, vol. 1(4), pp. 137–150.
11. Lesz S., Kalinowska-Ozgowicz E., Golombek K., Kleczka M.: Structure and properties of surface layers of selected constructional steels after sulfonitriding. Archives of Materials Science and Engineering, 2010, vol. 42(1), pp. 21–28.
12. Kula P., Pietrasik R., Pawęta S., Rzepkowski A., Filarczyk P., Komorowski J.: Technological equipment for use in the processes of producing low-friction layers on working surfaces arranged in a pile of piston rings. Patent Office of the Republic of Poland. Patent No. PL 429444, 2020.
13. Nikitin D.A., Nikitin P.D., Asoyan A.R., Solntsev A.A., Snarsky S.V.: Influence of the Piston Ring Shape in a Free State on the Efficiency of Sealing the Combustion Chamber, 2021, Intelligent Technologies and Electronic Devices in Vehicle and Road Transport Complex (TIRVED) – 2021, pp. 1–5, doi: 10.1109/TIRVED53476.2021.9639210.
14. ISO 6623:2013. Internal combustion engines – Piston rings – Scraper rings made of cast iron.
15. ISO 6624-1:2017. Internal combustion engines – Piston rings – Part 1: Keystone rings made of cast iron.
16. ISO 6626-2:2013. Internal combustion engines – Piston rings – Part 2: Coil-spring-loaded oil control rings of narrow width made of cast iron.
17. ISO 6625:1986. Internal combustion engines – Piston rings – Oil control rings.
18. Fan B., Feng S., Che Y., Mao J., Xie Y.: An oil monitoring method of wear evaluation for engine hot tests. The International Journal of Advanced Manufacturing Technology, 2018, vol. 94, p. 3199–3207, doi.org/10.1007/s00170-016-9473-8.