

Modification of Diffusion Layers by Laser Shock Peening

Jiri Prochazka (0000-0002-4894-5180)¹, Jindrich Vilis (0000-0003-1690-8454)¹, David Dobrocky (0000-0003-1400-3993)¹, Petr Sperka (0000-0002-4994-3144)²

¹Department of Mechanical Engineering, Faculty of Military Technology, University of Defence, Brno 662 10, Czech Republic. E-mail: jiri.prochazka@unob.cz

²Institute of Machine and Industrial Design, Faculty of Mechanical Engineering, Brno University of Technology, Brno 616 69, Czech Republic.

The article deals with a possibilities of an enhancement of functional properties of highly stressed components by specific combination of surface technology. Two surface technologies such as plasma nitriding and laser shock peening were selected for the experiment. Those technologies were applied upon steel 42CrMo4 frequently utilized in manufacturing of strained components. Properties obtained by applied surface technologies were tested by following experimental methods. The chemical composition was verified by optical emission spectrometer Tasman Q4 Bruker. The surface morphology was inspected by scanning electron microscope TESCAN MIRA 4. The microstructure of heat treated as well as of nitrated specimens was observed by opto-digital microscope Olympus DSX500i. The microhardness profiles were measured by microhardness tester LM247 AT LECO. The friction coefficient was tested on tribometer Bruker UMT-3 TriboLab. For an assessment of the surface wear-resistance the profilometer Talysurf CLI 1000 and Contour GT were utilized. The experimental results show that although the proposed surface technology combination manifests itself to be disadvantageous, both technology LSP, as well as plasma nitriding, applied separately, can lead to a significant wear reduction.

Keywords: Wear-resistance, Plasma nitriding, Laser shock peening, Highly stressed components

1 Introduction

Components of technology are exposed to many types of stress including tensile or compressive stress, bending, torsion, friction and wear, cyclic stress, corrosion, high temperatures etc., during their service life. A selection of components' material is influenced by a type of component significantly and also by a manner of its straining. In many cases of components exposed to combined stress not even the best material selection cannot fulfill all requirements placed on its properties. In such cases, a surface technology should be utilized.

By utilization of the surface technology it is possible to obtain a wide range of properties which differ from original properties of the component's material. Nowadays many sofistic surface technologies including for example Cold spray, Bimetal cladding, CVD or PVD coatings, chemical-heat treatment technologies etc., exist. In cases of the highly stressed components a surface hardening or case-hardening, due to their relatively cheap and simple application processes are still utilized dominantly. Those two surface technologies are introduced for a reason to create hard and wear-resistive surface layer, while the component's core remains tough. In both cases a martensitic transformation from an austenitic phase induced by rapid

cooling from temperatures around 900 °C is responsible for hardening of the surface [1], [2]. Unfortunately, during the rapid cooling a deformation or ruptures can occur. Thus, for a reason of a higher dimensional accuracy of the components, it is expedient to employ surface technologies of lower application temperatures. Two such technologies plasma nitriding and laser shock peening (LSP) were selected for the experiment.

In general, nitriding as well as a ferritic nitrocarburizing belongs to category of "low-temperature" chemical-heat treatment technologies. During the process, at temperature chosen from range 300 - 580 °C, nitrogen is introduced into the surface microstructure from nitriding atmosphere [3], [4]. The nitrogen creates hard nitrides and carbonitrides with iron and mainly with some alloying elements such as Cr, Mo, Al, V, W etc. which results in a creation of hard and wear-resistive surface layer [5]. Although the stable nitriding industrial process has been known since the middle of the twentieth century, the nitriding process, materials predetermined for nitriding as well as new approaches for industrial nitriding utilization are developed continuously [6]. One such approach is based on a creation of hybrid surface treatment combining nitriding with another surface technology. The most recent researches are focused on a creation of duplex coatings created by hard PVD or PACVD

coatings in combination with diffusion layers such as nitride layers [7]. In this experiment a different surface technology such as LSP in combination with plasma nitriding was utilized.

LSP is surface technology based on a mechanical surface hardening caused by shock waves induced by effect of laser beam pulses. This technology results in creation of a surface layer which provides a higher microhardness, a higher wear-resistance and also a corrosion resistance [8]. In addition, due to an existence of a residual compressive stress in the layer, the treated surfaces are less prone to fatigue cracking, which is the most common reason for using the LSP. In case of standard LSP technology the laser beams impinge on the treated surface close to each other or can be overlapped. Hence a whole area of the surface is affected by the treatment.

In this manuscript an another possible advantage of LSP in combination with nitride layer which may prolong service life of the highly stressed components was tested. Contrary to standard technology, much wider spacing between individual laser beams have been set. This adjustment should result in a creation of “dimpled surface”. Dimples caused by impacts of

laser beams on the treated surface can serve as reservoirs of lubricant or can act as a spaces where impurities and wear debris can be washed away from contact surface. Hence, the aim of this manuscript is to verify, whether the wear-resistance of the surface by the selected treatment can be increased.

2 Materials and methods

Based on analysis of the materials frequently utilized in manufacturing of the highly stressed components the steel 42CrMo4 was selected for the experiment. The steel can be found in components such as crankshafts, camshafts, parts of weapons as well as in highly precision components of ball-screws [9], [10]. Hence, within the experiment, properties obtained by application of plasma nitriding and LSP upon the steel 42CrMo4 were tested.

2.1 Specimen preparation

From the steel bar of diameter 65 mm obtained in normalized state four discs of thickness 7 mm were cut-off. The discs were heat treated subsequently in compliance with parameters listed in Table 1.

Tab. 1 Heat treatment parameters

Quenching			Tempering		
Temperature [°C]	Time [min]	medium	Temperature [°C]	Time [min]	medium
850	20	water	600	60	water

For a reason of possibility to mount the specimens in the tribometer, two holes were drilled into the discs. Then, horizontal surfaces of the discs on the instrument BPH 300 with requirement for parameter $R_a < 0.4 \mu\text{m}$ were ground. Two specimens were let in the heat-treated state. Two remaining specimen-temperature 540 °C to plasma nitriding were subjected. The s are shown in Figure 1.

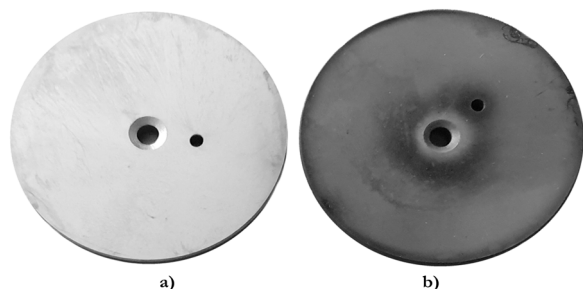


Fig. 1 Experimental specimens; a) Heat treated; b) Plasma nitrided

All specimens were sandblasted with Al_2O_3 of grains from 280 μm to 420 μm under pressure 0.3 MPa subsequently. Finally, the LSP with spacing of laser beams impacts 150 μm was applied upon one heat-treated and upon one plasma nitrided specimen. The specimens thus prepared were utilized for experimental measurements.

After the experiment, in order to create metallographic samples, a small segment of material from outer edge of each specimen was cut-off. The segments were molded into a thermoplastic powder, grinded and polished with velvet by employing paste with diamond grains of size 0.5 μm .

2.2 Chemical composition

The chemical composition of the steel 42CrMo4 by optical emission spectrometer Tasman Q4 Bruker was verified. Results of the measurement are listed in Table 2.

Tab. 2 Chemical composition of steel 42CrMo4 (wt. %)

C	Mn	Si	Cr	Mo	Ni	P	S
OES							
0.39	0.87	0.36	1.12	0.23	0.25	0.005	0.017
Standard							
0.38 – 0.45	0.60 – 0.90	< 0.40	0.90 – 1.20	0.15 – 0.30	< 0.50	< 0.025	< 0.035

The table implies, that content of chemical elements contained in the steel 42CrMo4 is in compliance with its material datasheet. According to the content of chromium (1.12 wt. %) nitrides and carbides creating element, creation of hard and wear-resistant nitride layer by application of plasma nitriding can be expected.

2.3 Surface morphology

The surface morphology was observed primarily for a reason to verify, whether the surface morphology of the heat-treated and the nitrided specimens is the same, or different (Figure 2). For observation the scanning electron microscope TESCAN MIRA 4 was used.

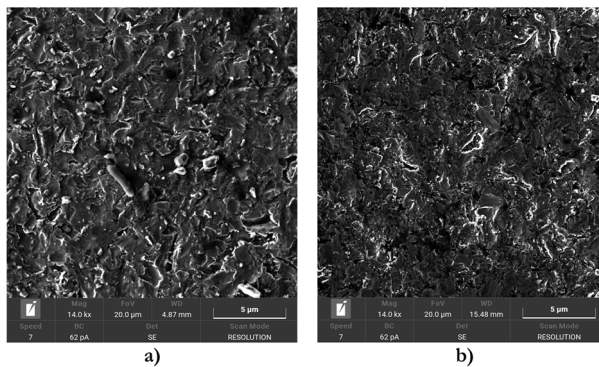


Fig. 2 Surface morphology of sandblasted specimens magnified 14 000x; a) Heat treated; b) Plasma nitrided

A minor differences between surfaces of the heat treated and the plasma nitrided specimens after sandblasting are visible. In case of surface of the plasma nitrided specimen, craters induced by impacts of abrasive grains within sandblasting are shallower and less

ragged contrary to the heat treated specimen. It can be caused by a higher surface hardness of the nitrided specimen contrary to the heat treated one. By the roughness measurement only a negligible deterioration of a hundredth of Ra were found, thus the requirement for $Ra < 0,4$ has been also met.

In the Figure 3 a contrast between sandblasted surfaces with and without application of LSP at magnification of 500x is visible.

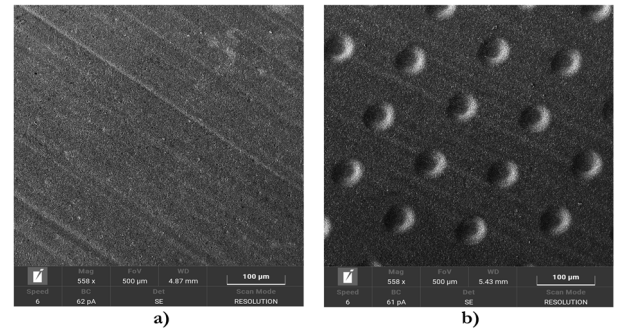


Fig. 3 Surface morphology of sandblasted specimens magnified 500x; a) Heat treated; b) Heat treated + LSP

The Figure 3b shows the dimpled surface of the heat treated specimen after application of modified LSP technology. As it has been expected, by setting of wider spacing of laser beams only a part of the surface area was affected.

2.4 Microstructure

The microstructure of the heat treated specimen as well as of the nitride layer were observed by the Opto-digital microscope DSX500i (Figure 4).

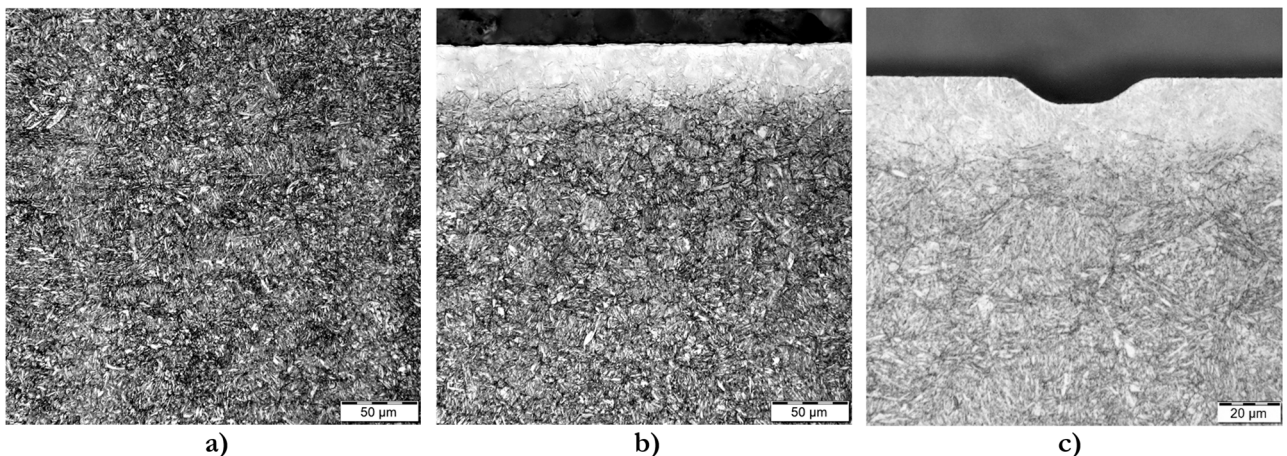


Fig. 4 Microstructure; a) Heat treated specimen; b) Nitride layer; c) Nitride layer + LSP

The microstructure of the heat treated specimen's core visible in Figure 4a is in accordance with the heat treatment parameters. The Figure 4b shows a cross-section of a typical nitride layer composed of three areas which include a compound layer on the top of

the surface, a diffusion layer situated under the compound layer and a transition area in the middle of the diffusion layer and the original microstructure [11], [12]. In this case the compound layer is barely visible due to the sandblasting by which the compound layer

was almost removed. The last figure 4c shows the cross section of the dimple induced by the LSP technology upon the nitride sample. No additional microstructure changes induced by LSP are visible in the figure.

2.5 Microhardness measurement

The most important area of the nitride layer is the diffusion layer composed of the original microstructure and a dispersion of nitrides as well as carbonitrides of alloying elements. Resulting properties of the nitrided surfaces particularly depend on maximum values of the microhardness as well as on nitriding hardness depth. The nitriding hardness depth (NHD) is defined by standard ISO 18203:2016 [13]. The NHD parameter can be measured by utilizing the microhardness profiles. The profile is obtained by consequence of microhardness measurements performed on a cross-sectional specimen by Vickers or Knoop indenter loaded by force chosen from range 0.9807 N – 9,807 N. The first impression should be made very near to the surface in distance only 2.5 times greater than an impression's diagonal. Following impressions are led normal to the surface in direction inwardly to the material. A vector of impressions setting in the Cornerstone software, by which the utilized automated microhardness tester LM247 AT LECO is equipped, is visible in the figure 5.

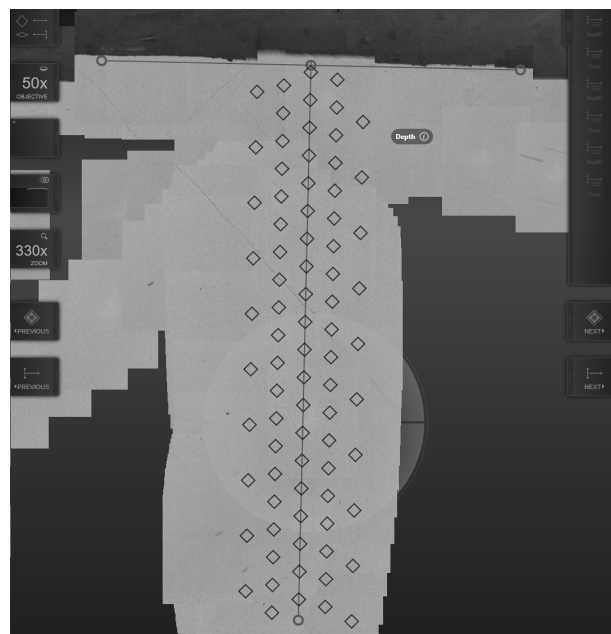


Fig. 5 Vector of impressions

With increasing distance from the surface the microhardness values decrease. By plotting data such measured into dependence of microhardness upon depth, the microhardness profile is created. For the reason to determine NHD parameter, a core microhardness must be measured additionally. Then, NHD is defined as a distance between surface and a depth,

where the core microhardness increased by 50 HV was measured.

2.6 Friction coefficient measurement

The friction coefficient is an essential tribological parameter defined as quotient of tangential force and normal force. In the experiment, the friction coefficient in accordance with the Ball-On-Flat experimental method described in the standard ASTM 133-05 by tribometer Bruker UMT-3 TriboLab was measured [14]. Ball indenter is forced to the specimen's surface by a predetermined load while the specimen mounted to a support moves reciprocally. Both contact surfaces i.e. surface of the ball indenter and of the specimen adapt mutually during the measurement which has an impact upon values of the friction coefficient. Hence, within measurement, the values are recorded continuously and plotted into graph in dependence on time.

Four phases of wear such as wear initiation, contact surface adaptation, friction coefficient stabilization and a surface layer destruction can be distinguished in the graph. The first two phases together are also known as "running-in" phase. Wear debris generated on the contact surfaces can be stucked in the wear path and thus, a thin film composed of wear debris and oxides can be created. Hence, contrary to the third phase, the running-in phase by a higher values of friction coefficient is accompanied. After that, when the film in the wear path is created, the friction coefficient values are stabilized. This phase, also called steady-state takes much longer compared to previous phases. The last phase, typical for a surface destruction, by a rapid increase of friction coefficient manifests.

2.7 Surface wear-resistance assessment

The last step of the experiment was an assessment of the surface wear-resistance. The surface wear-resistance based on wear occurred in tribological wear paths created by standard Ball-On-Flat method were assessed. Qualitative assessment based on wear paths observation by Opto digital microscope DSX500i was performed. For a quantitative assessment the parameters of cross-sectional profiles of the tribological wear paths obtained by profilometer Talysurf CLI 1000 as well as 3D surface scans taken by Contour GT were utilized.

3 Results and discussion

All above mentioned experimental methods upon heat treated as well as upon nitrided specimens with and also without LSP were applied.

3.1 Microhardness profiles

For the measurement the automated microhardness tester LM247 AT LECO in accordance with

ISO 18203:2016 was utilized. Parameters of the measurement was set as follows: Vickers' indenter, load

0.9807 N (i.e. HV0.1), spacing 10 μm . The microhardness profile of the plasma nitrided specimen is shown in the Figure 6.

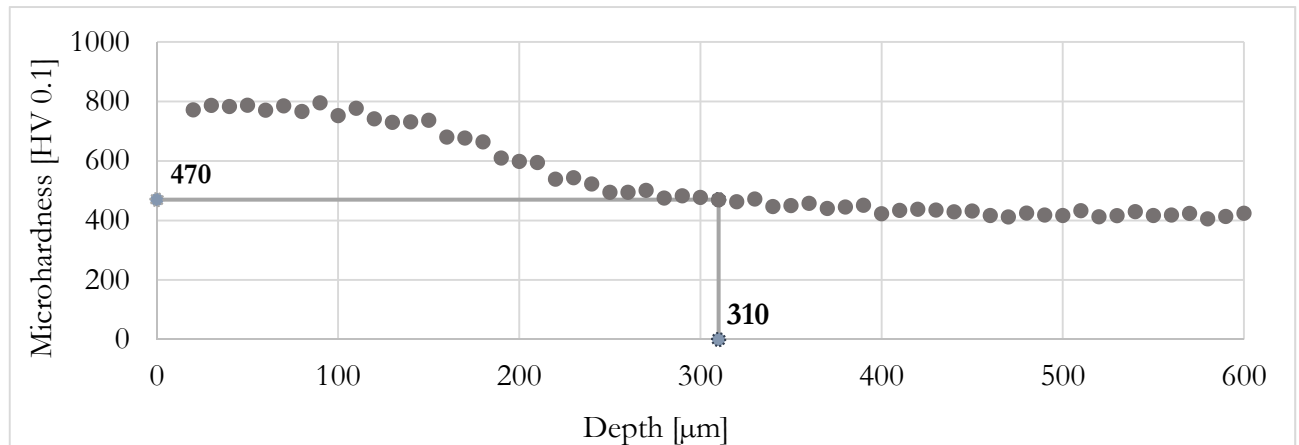


Fig. 6 Microhardness profile

From the profile follows, that microhardness approximately 800 HV0.1 in a vicinity of the surface by the plasma nitriding has been reached. The microhardness of the core as an average value from three measurements to 420 HV0.1 was determined. Hence, NHD = 310 μm for the microhardness limit 470 HV0.1 (420 HV0.1 increased by 50 HV0.1) was defined.

3.2 Friction coefficient

The friction coefficient of tested specimens at load 15 N within 1000 seconds at room temperature and with frequency 5 Hz was measured. The measurement was performed threetimes on each specimen. The representative results are visible in Figure 7.

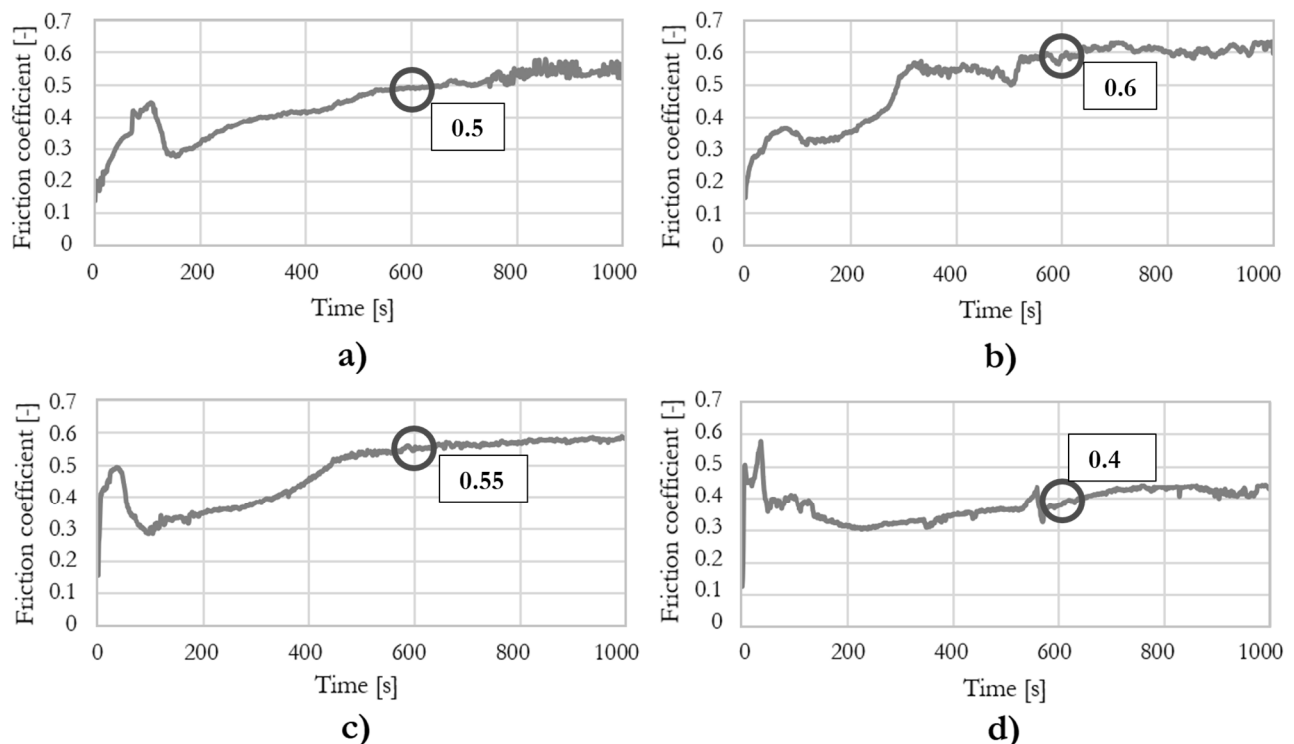


Fig. 7 Friction coefficient; a) Heat treated; b) Plasma nitrided; c) Heat treated + LSP; d) Plasma nitrided + LSP

In all cases, steady-state wear was reached by the 600th second. By comparison of the graphs 7 a) and 7 b) can be stated, that nitride surface of the steel

42CrMo4 exhibits a higher friction coefficient than solely heat treated specimen. The phenomenon can be attributed to the hard particles, especially nitrides and carbonitrides present in the tribological wear path.

These particles can be stacked in the wear path which can cause deterioration of the sliding condition. Thus, the friction coefficient values increase. As it is described in literature e.g. [15], the increase of friction coefficient values after plasma nitriding is not unique for the tested steel, but it is typical for nitriding in general.

In graphs a) and c) in Figure 7, there are no significant differences in the friction coefficient values. Hence, dimples created by LSP have almost no effect upon friction coefficient of the heat treated specimen.

However, a positive effect of the LSP technology on the coefficient of friction was observed in the case of the nitrided specimen. While the nitrided surface

itself showed an increase in friction coefficient values compared to the heat treated surface, the subsequent application of LSP after nitriding resulted in a slight decrease in coefficient values. It can be related to a possibility of the hard particles to be trapped in the dimples created by the LSP technology. A lower amount of hard particles and other wear debris in the tribological wear path leads to the friction decrease.

3.3 Wear-resistance

The tribological wear paths created within the friction coefficient measurement which are visible in Figure 8 were also utilized for the assessment of wear-resistance.

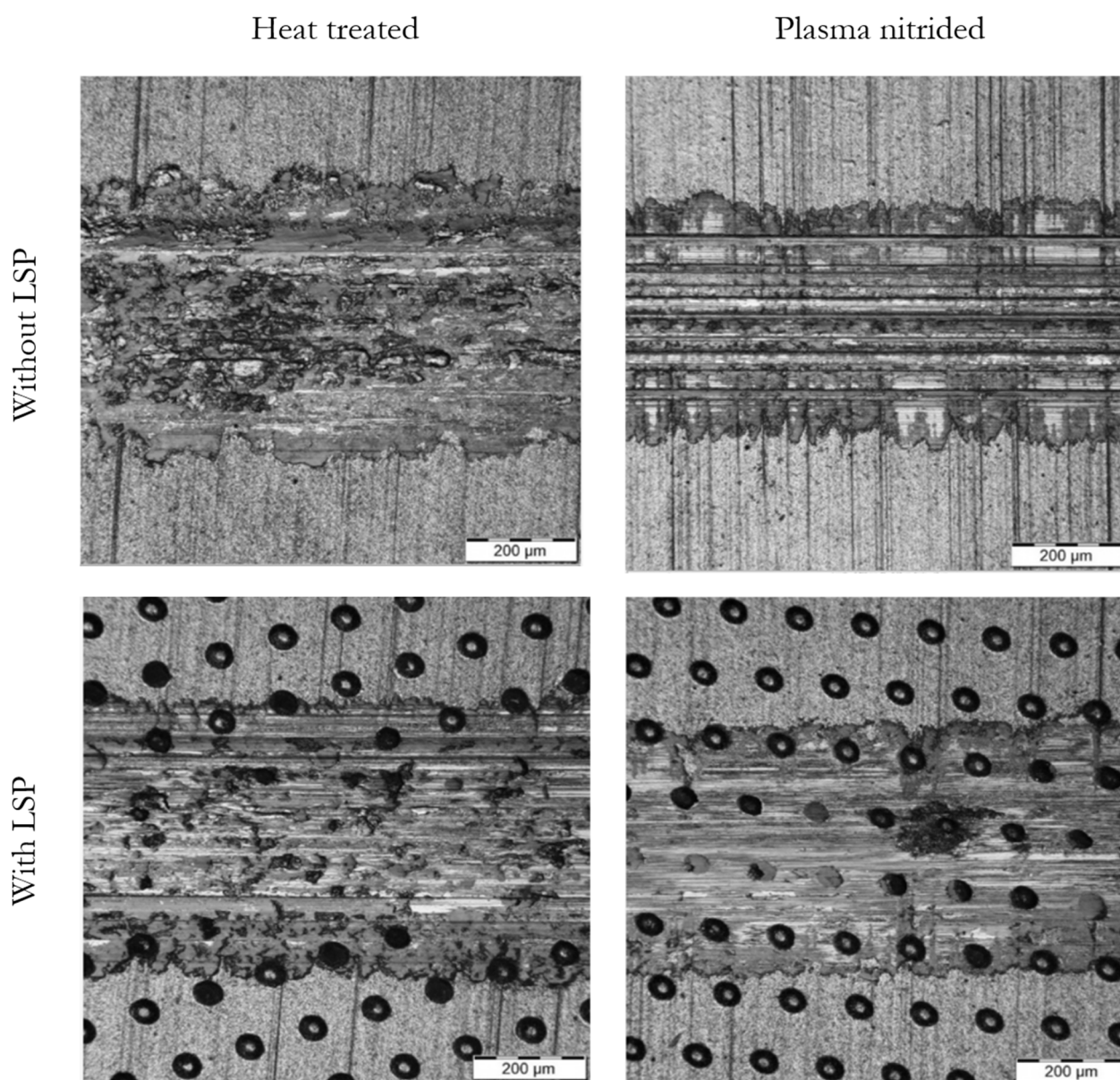


Fig. 8 Tribological wear paths

Observation of the Figure 8 implies, that the heat treated specimen's surface is more prone to wear contrary to the nitrided specimen. The phenomenon is closely related to presence of the nitride layer which is

harder than solely heat treated surface which was demonstrated by the Figure 6. While similar wear paths are visible in case of both heat treated specimens (i.e. with and without LSP), the tribological wear paths

of the nitrided specimens differ significantly. The wear path obtained on the laser shocked peened nitrided surface is much smoother without longitudinal scratches contrary to the nitrided specimen without LSP.

From the observation flows, that dimples created by the LSP technology upon nitrided specimen are less susceptible to crumble contrary to dimples on the heat

treated specimen. Thus, wear debris can be gathered in the dimples much longer during the measurement.

Wear of the specimens was also assessed quantitatively. For the assessment the tribological wear paths cross-section profiles as well as their 3D scans were utilized. Average values of the results of three measurements performed on each specimen are listed in Table 3.

Tab. 3 Wear of the specimens

Technology	Wear (μm^3)	Coefficient of variation
Heat treated	$85.8 \cdot 10^5$	0.061
Heat treated + LSP	$66.2 \cdot 10^5$	0.086
Plasma nitrided	$17.5 \cdot 10^5$	0.103
Plasma nitrided + LSP	$46.3 \cdot 10^5$	0.154

The greatest wear around $85 \cdot 10^5 \mu\text{m}^3$ in case of the heat treated specimen was found. By application of LSP technology upon the heat treated specimen a minor wear-resistance improvement approximately by

20 % was achieved.

In case of the nitrided specimen the lowest wear was measured, which is visible in Figure 9.

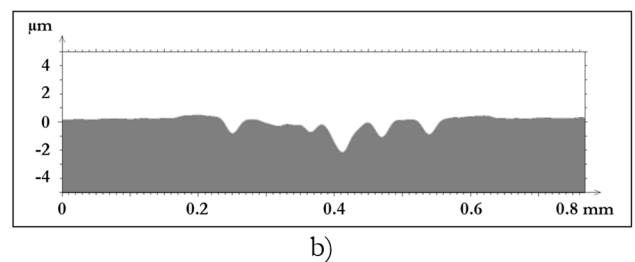
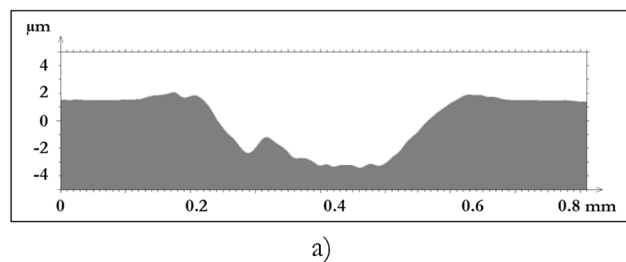


Fig. 9 Cross-sectional profiles of wear paths; a) Heat treated; b) Plasma nitrided

Due to presence of hard and wear-resistive nitride layer created by plasma nitriding the wear was reduced by approximately 80 % in comparison with solely heat treated specimen. Moreover, for a reason to further improvement of wear-resistance of the nitrided surfaces it is also possible to combine it with PVD coatings, as it is described in literature e.g. [16].

Contrary to heat treated specimen, in case of the plasma nitriding a positive effect of LSP upon surface wear-resistance at solid sliding was not confirmed. Moreover, the wear-resistance after application of the LSP technology upon nitrided surface was deteriorated. However, the wear of the nitrided specimen treated with LSP was also reduced by 50 % in comparison with the heat treated specimen.

4 Conclusion

Tribological surface properties of specimens treated by different technologies in the manuscript were assessed. By employing Ball-on-Flat experimental method at room temperature following results were found. By application of plasma nitriding upon the heat treated specimen the values of friction coefficient

were increased. This phenomenon can be attributed to a presence of hard wear particles such as nitrides and carbonitrides which act abrasively during the measurement. On the other hand, the results pay primarily in the case of room temperature. In case of a higher temperature, for example at 80 °C, the nitrided surface is more prone to a creation of an oxide layer which may cause a decrease in values of friction coefficient [17]. After application of the LSP technology upon heat treated specimen a minor deterioration of the friction coefficient was also found.

Although after application of plasma nitriding upon heat treated specimen the friction coefficient was increased, a presence of dimples created by LSP technology on the nitrided surface subsequently caused a significant reduction in values of friction coefficient. Thus, it can be stated, that combination of plasma nitriding and LSP technology seems to be advantageous in cases where friction coefficient need to be reduced.

The assessment of the specimens' surface wear-resistance showed that application of LSP upon solely heat treated specimen led to a minor reduction of wear

approximately about 20 %. This result is in compliance with literature e. g. [18].

Although in case of the plasma nitrided specimen a capture of a wear debris in dimples created by LSP in the Figure 8 is visible, significant wear-resistance deterioration in comparison with solely nitrided specimen was found. Hence, in case of wear-resistance, the combination of LSP with plasma nitriding seems not to be as advantageous as it has been presumed. Thus, based on the results of the experiment, for a reason of wear-resistance improvement this combination can not be recommended. However, contrary to LSP, by the application of plasma nitriding upon heat treated specimen a significant reduction of wear approximately about 80 % was reached. The wear reduction is connected with the presence of the hard and wear-resistive nitride layer, of which depth and maximum microhardness values by the microhardness measurement were determined as 300 μm and 800 HV0.1, respectively.

Based on the experimental results, it can be concluded that LSP as well as plasma nitriding technology can be applied separately for a purpose to wear reduction.

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