EFFECTS OF PLASMA NITRIDING AND SURFACE DENSIFICATION ON CONTACT FATIGUE OF ASTALOY CRM BASED PM STEEL

D. Rodziňák, J. Čerňan, M. Lengyel, R. Zahradníček

Abstract
The effects of plasma nitriding of Astaloy CrM + 0.3% C sintered material with and without a shot peened surface on contact fatigue were studied. Tests were conducted on a "ball on washer" system and evaluated by a classical Wöhler curve to the value of $50 \cdot 10^6$ cycles. The results showed a very positive influence of the simultaneously acting reinforced by shot peening material surface (additionally with reduced porosity in this area) and chemical-thermal treatment by plasma nitriding. The fatigue strength for lifetime of $50 \cdot 10^6$ cycles increased from the value of 1200 to 2400 MPa. Microscopic study showed the different mechanisms of crack formation and of crack propagation rate due to contact loading of material with a shot peened surface, which has an obvious impact on material lifetime.
Keywords: sintered Cr steel, contact fatigue, shot peening, plasma nitriding

INTRODUCTION

Material durability is of constant interest for many scientific studies. One of the factors limiting lifetime, of certain machine parts and equipment in interactive mechanical contact, is damage by one of the manifestations of tribology. Such damage must be eliminated as thoroughly as possible. Classical methods applicable in this case include surface treatment of materials by a chemical-thermal processing such as carburization and nitriding, and also mechanical methods such as shot peening and rolling.

These processes are successfully used not only for fully dense materials, but also in the case of sintered materials. PM materials have different basic characteristics and therefore the use of surface treatment techniques, though possible, leads to different quantitative results. An example could be the influence of shot peening on the properties of sintered materials, e.g. hardness, porosity, contact fatigue. The results we achieved in [1] show that shot peening at Astaloy CrL and Astaloy CrM with 0.3 / 0.7% graphite, increased the surface hardness of HV 10, compared to the sintering state, by about 100% and the fatigue limit by 4-18%. Increasing the density in the surface layer from 7.00 to 7.45 $g \cdot cm^{-3}$ is also important. It is therefore necessary to subject the PM materials to these technologies and map their behaviour and determine the conditions under which their use is acceptable. Their use is known from the literature [2-5], including work of our own [6-7].

Currently there is a boom in the use of new modern technologies, such as different surface treatments in a vacuum or with inert gases. These methods are currently of great research interest in the field of sintered materials in connection with their possible use in the manufacture of demanding components exposed to tribological damage (friction,

Dušan Rodziňák, Jozef Čerňan, Martin Lengyel, Rudolf Zahradníček, Faculty of Aeronautics, The Technical University of Košice, Košice, Slovak Republic

contact fatigue) [8-14]). The method of plasma nitriding is also ranked among these technologies. Its influence on properties of sintered material of the type Astaloy CrM (commercial prealloyed iron based powder from Höganäs AB, Sweden) in connection with contact stress is the topic of this contribution.

EXPERIMENTAL

Prealloyed powder Astaloy CrM (Fe - 3% Cr - 0.5% Mo, Höganäs AB) was used as the base powder for specimen preparation. After the addition of graphite of 0.3% and 0.7% and a HWC type lubricant, the powder mixtures were compacted at a pressure of 600 MPa to dimensions of Ø 30 x 5 mm. Compacted specimens with "green strength" were subsequently sintered at 1120°C for 60 min in 90% N_2 + 10% H_2 atmosphere. To prevent a possible oxidation of the specimens, the atmosphere was frozen (dew point -57°C) and also the specimens were put into a retort with a powder mixture of Al_2O_3 and 5% C. After sintering, the specimens were cooled in a retort in a protective atmosphere outside the furnace.

The specimens produced in this way were subsequently machined to the outer diameter of 28 mm and the internal bore of Ø 10 mm. Both sides of the specimen surface were ground, mainly to achieve flatness, which is very important for a balanced load and for dimensional tolerances. Half of the specimen of each material was subjected to shot peening on both surfaces. The process of surface hardening was carried out by shot peening iron granules Ø 0.6 mm at a speed of 7000 rpm and at an angle of 90° to the surface of the specimen. On both sides of the specimen 9 kg granulate was used. Subsequently, all specimens were subjected to plasma nitriding on both sides (the work of the Academy of Defence, Brno) under the following conditions: nitriding temperature of 500°C; 24:8 ratio of hydrogen gas, nitrogen pressure of 2.8 mbar, 520 V voltage, pulse duration and pulse spacing of 100 ms; nitriding time of 35 hours.

The specimens were subjected to contact fatigue tests on the device type AXMAT (Fig.1), which operates as "ball on washer" tester, i.e. sphere on plane geometry, with a frequency - $500 \cdot s^{-1}$. Between the apparatus bearing ring and the specimen ring, a cage of 18 balls with a diameter of 3.969 mm made from conventional bearing steel – DIN 100 Cr6 (62 HRC) was positioned. The analysis is the same as for the more commonly used "pin on disc" apparatus. The balls are lubricated by transmission oil SAE 80th MOGUL. The oil circulating in the tests was constantly filtered. The results of contact fatigue were evaluated by a classical Wöhler curve to the value of the life cycles of $50 \cdot 10^6$.

The Hertz stress to which the specimens were subjected were calculated by the equation

$$\sigma_{\max} = 0,388 \cdot \sqrt[3]{4 . F . \frac{E_1^2 . E_2^2}{(E_1 + E_2)^2} . \frac{1}{R^2}}$$

where F - the applied force, R - radius of the ball, E_1 - modulus of elasticity of steel balls – 210 GPa, E_2 - modulus of sintered material. The modulus of sintered material was calculated according the equation $E_2 = E_1 . \left(\frac{\gamma_2}{\gamma_1}\right)^{3.4}$ [15], where γ_1 and γ_2 are densities. For density $\gamma = 7.0\ g \cdot cm^{-3}$ it is 142 GPa. In the case of a material whose surface has been shot peened, density in the surface layers increased to 7.45 -7.5 $g.cm^{-3}$, which according to the equation [13], corresponds to $E_2 \sim 180$ GPa. Density in the surface layer was computed on the basis of measuring and comparing the metallographical images in non-etched states of surface porosity of both materials (the shot peened and not shot peened).

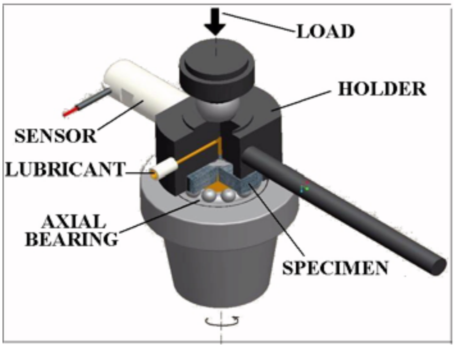


Fig.1. Principle of contact fatigue test.

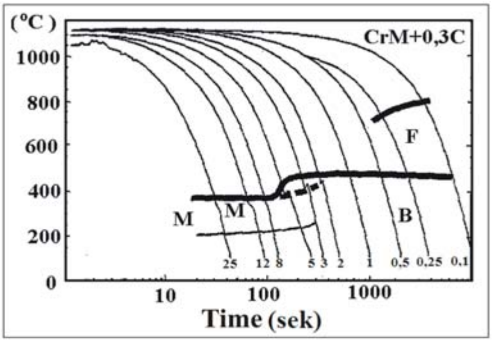
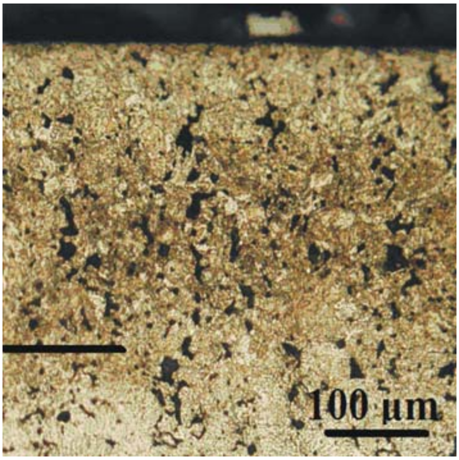


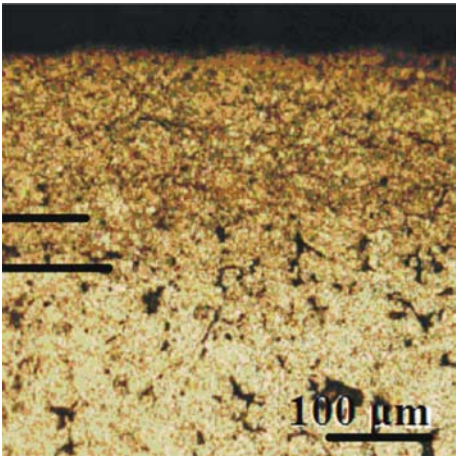
Fig.2. Effect of cooling rate on structure.

Mechanical properties of the specimens were measured to enable their comparison with the literature. Hardness was measured by Vickers HV10 and Rockwell HRB methods. The influence of shot peening was assessed on the basis of metallographic sections of the specimen thickness using an optical microscope. Nitriding layer thickness was determined from the specimen metallographic sections perpendicular to the specimen axis by measuring the Hanneman microhardness on optical microscopes Neophot 21. Metallographic specimens of all materials were made by the classical manner (embedding in dentacryl, grinding, polishing, etching - Nital) and were used for the study of microstructures, as well as for the assessment of cracks generated during contact fatigue. After contact fatigue testing, the traces of circulating balls were documented and pitting sizes were measured, depending on the driven cycles and the Hertz stress. Moreover, the wear for the material traces was measured by roughness measurement using the SURFTEST Mitutoyo SJ profilemeter.

RESULTS

The material Astaloy CrM + 0.3% C had a bainitic microstructure, as illustrated in Fig.3. This is also consistent with the cooling rate, which was given by conditions after nitriding. Based upon and from the microstructure character, which can be seen in Fig.3a, we can conclude that it is upper bainite. Figure 3b, where the specimen surface is shot peened, shows the depth of the hardening, which is manifested by an increased density (absence of pores). A depth of approximately 150 μm was determined.





(a) (b)

Fig.3. Material microstructure of Ast CrM + 0.3% C in the state without (a) and after shot peening (b).

The plasma nitriding treatment for the two materials created nitrided layers on the surfaces. In this layer the chemical composition is changed, which is manifested by an increased etching ability. On this basis, we can determine the nitriding depth by measuring this value by microscopic observation (see the parallel lines in Fig.3). It was set to 300 µm. The effect of shot peening was reflected in depth, which is determined by the upper short line - Fig.3b and the depth value is about 150 µm. The effect of nitriding expressed by a higher etching ability increased in this case to a depth of 235 µm. It follows from this that by shot peening the material, resulting in subsurface layers compressed by plastic deformation, the penetration of the nitriding effect is reduced to 235 µm from 300 microns in specimens without shot peening. In addition, on the surface a very thin, so-called ε - layer - Fig.4 is created, with a thickness of about 3-4 µm. In our case, however, it does not play a dominant role, so no further attention will be paid to it. Its importance is greater in the case of testing for abrasive wear. Those results are the subject of another article which will also be published in Powder Metallurgy Progress.

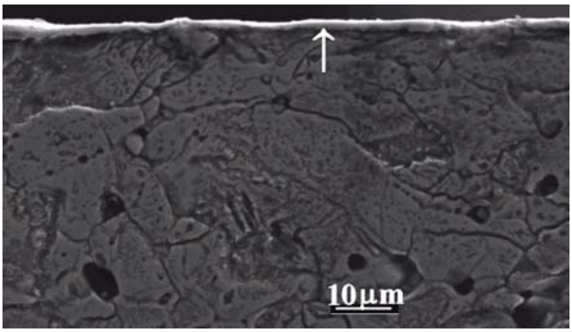


Fig.4. ε – layer on surface after plasma nitriding.

Moreover, it is possible to detect hardening of the surface layer by measuring the microhardness of the specimen cross section. In the specimen without shot peening, microhardness determined the influence of nitriding and its depth. For shot peened specimens, the additive impact of blasting and nitriding was measured. To determine this value the minimum depth of influence was set at 500 HVM 50 (this value is indicated by a horizontal line in the diagram in Fig.5). The line intersects the two curves at the values which are in accordance with the values that we have measured on the microscopic analysis basis.

Besides this, it is possible to precisely determine the depth of nitriding on the basis of fracture-micro-structural characteristics. This method was also used here, and the results correspond to earlier ones. A more detailed description and documentation is available in our previous work, e.g. [7].

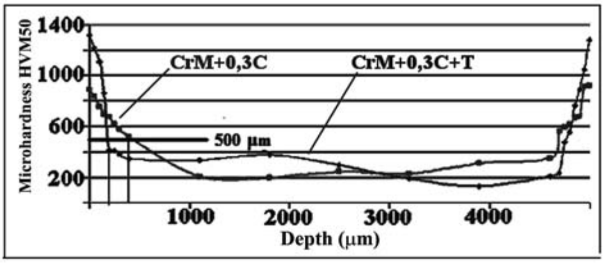


Fig.5. Microhardness variation in cross-sections of specimens.

To compare the properties of this material after treatment, the macrohardness on the specimen surface was measured using Vickers and Rockwell methods. Table 1 shows how the different steps in the processing technology of the samples resulted in actual hardness.

Tab.1. Surface hardness and fatigue life of materials.

	HV 10	HRC / HRB	σ_C [MPa]
Sintering	200	82	1154
Sintering+shot peening	440	20 102	1200
Sintering+ plasma nitriding	560	29	1200
Sintering+ shot peening+ plasma nitriding	860	31.5	2400

Contact fatigue results, presented in Tab.1 and in Fig.6, show that plasma nitriding of material Ast CrM + 0.3% C (for $50 \cdot 10^6$ cycles) reached a value of 1200 MPa. This value is slightly larger than that achieved in our previous research, e.g. [12] on nitrided material, 1154 MPa. It is caused by nitriding, which did not affect porosity, but had a great influence on brittleness. For specimens which were shot peened after sintering [12], the increase was from 1200 to 2400 MPa: it represents an increase of 100%.

In addition, the results show that the impact of shot peening and plasma nitriding is essentially the same in terms of fatigue life for the value of $50 \cdot 10^6$ cycles. The actual curve for the nitrided condition, however, lies under the curve for shot peening status. Comparing the values of fatigue life with the values of hardness, we can see that the plasma nitriding increases the surface hardness significantly higher than does plastic deformation.

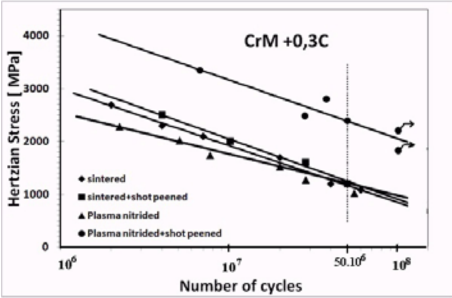


Fig.6. Wöhler curve for contact fatigue tests.

This apparent difference lies in the processes which in the material are activated by plasma nitriding and plastic deformation by shot peening. Plastic deformation by shot peening causes strain hardening on the material surface. It results in an increase of surface hardness values from 200 to the value 440 HV10. In addition, there is an increase of surface density. And it is also manifested by pressure of residual stresses formed on the surface. Plasma nitriding increases hardness compared to the sintering condition of 560 HV10.

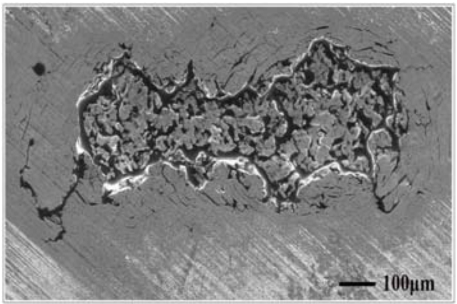


Fig.7. Pitting on Ast CrM + 0.3% C material.

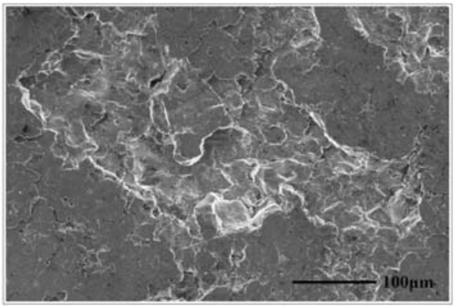


Fig.8. Pitting on Ast CrM + 0.3% C + Sp material.

Furthermore, nitriding pressure causes residual stresses in the surface layer, though these are small. According to the authors [14], gas nitriding itself at 480°C / 1 hour produces surface compressive stress of 50 MPa. As mentioned above, both surface treatments separately provide the same result for fatigue life of $50 \cdot 10^6$ cycles, namely the value of 1200 MPa. The main cause of this are contact fatigue damage mechanisms as shown in Fig.7 for the nitrided material Ast CrM + 0.3% C and Fig.8 for material Ast CrM + 0.3% C shot peened before nitriding.

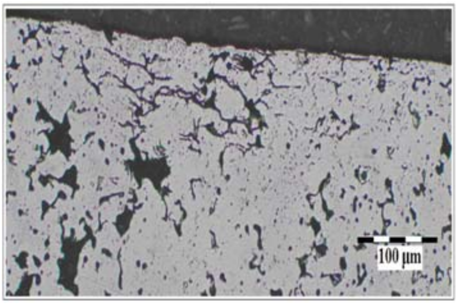


Fig.9. Development of cracks in the track in material without shot peening.

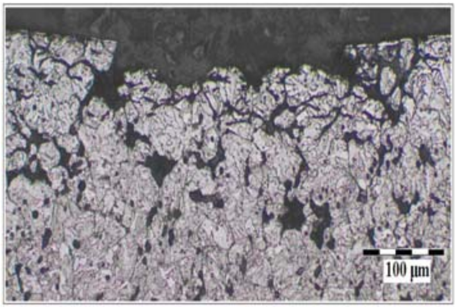


Fig.10. Cross-section for pitting for the shot peened state.

In the first case cracks can be seen around the pitting, which tell us that their orbiting ball pressure generates tension in surface layers, causing cracking. Their combinations result in the loss of material particles, which ultimately create pitting. Nitriding creates a hard surface layer of nitride, including a white ε-layer. We can say that the nitriding, though increasing hardness and wear resistance, also turns the material brittle. In our previous work [7], we documented the fact that the sintered material is damaged by ductile dimple morphology. Nitrided material exhibits brittle fracture with cleavage facets. When under contact fatigue loading, the brittle material on the surface is not capable of plastic deformation. At the interface of hard particles (nitrides, carbides, oxides) and matrix, due to differences in plasticity, the creation of crack nuclei occurs. This fact must be added by the impact of stress concentrations between the pores, giving rise to a large number of small cracks and at the same time also affecting the size of pittings. They are small, and their subsequent gradual bonding creates the appearance presented in Fig.7.

This fact is confirmed by the results given in [14] where the authors have documented the presence of a white layer on the surface of pores inside the material. It can be concluded that cracks arise and grow mainly at depths of Hertzian stress peaks in Figs.9 and 10 (non-etched and etched condition). Cracks run more or less parallel to the surface at depths that can be determined both by the scale shown on both photographs and measuring the roughness of the surface passing through the track - Fig.11, and in places where pitting is contained in the trace - Fig.12. As shown by these measurements, the depth of pitting was to the order of 40-50 µm which corresponds to the depth of maximum Hertzian stress for this case (for 2270 MPa stress – the calculated depth is of max. 0.048 mm). A similar mechanism applies only to the shot peened state. The material densifies by plastic deformation, further stress causes the generation of cracks at the interface between the hard particles (mostly between oxides) and the matrix, or between two pores. Both of these effects (deformation and nitriding) separately are the cause of the fact that contact fatigue life does not relate to the hardness values. Low value of the fatigue life after nitriding was observed by other authors as well [13].

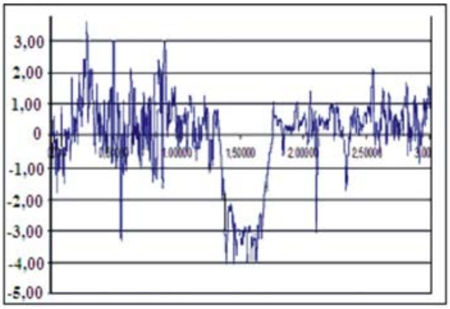


Fig.11. Profilograph of a track.

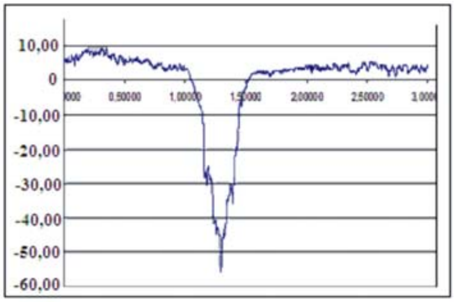


Fig.12. Profilograph of pitted site.

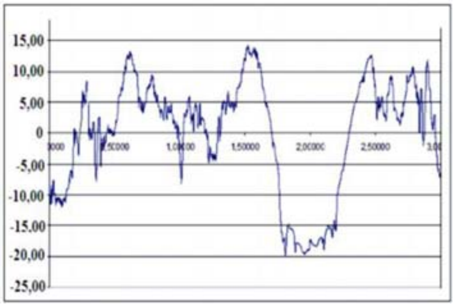


Fig.13. Profilograph of pitting site of shot peened material.

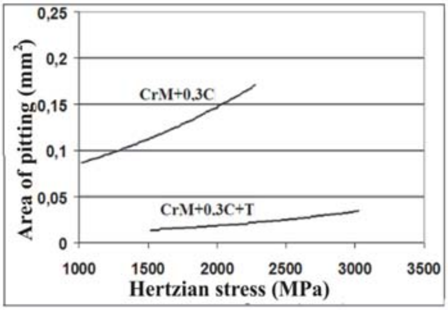


Fig.14. Size dependence area of pitting on the Hertz stress.

In the case of shot peening, before nitriding plastic deformation by surface compaction (absence of pores) occurs. Although strain hardening due to shot peening was eliminated by the temperature of nitriding, 500°C / 35 hours, the compacted layer will reduce the number of pores, increasing the distances between them (reducing the impact of stress concentrations) and simultaneously it makes it more difficult for the nitridating to penetrate into the core of sample materials (see Fig.3a and 3b). This fact, coupled with high surface hardness (860 HV10) in contact fatigue loading, then increases the need for higher stress to cause cracks and their propagation. This results in a synergistic effect manifested by an increase in fatigue life at 2400 MPa.

This fact is also reflected in the appearance of pitting – Fig.8. It consists of small extracted particles and its depth is much smaller. Measurement of the parameters of this type of pitting has shown - Fig.13 that the depth was about 10 to 15 μm. Plastic deformation in the surface layers causes the generation of cracks - as is illustrated in Figs.15 and 16. It is also evident that the cracks originate at the surface and spread beneath the surface at places of maximum deformation. These, by their shape, correspond to the impact of metal granulate.

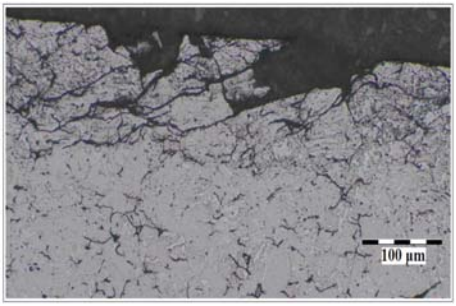


Fig.15. Crack creation in a track of shot peened material.

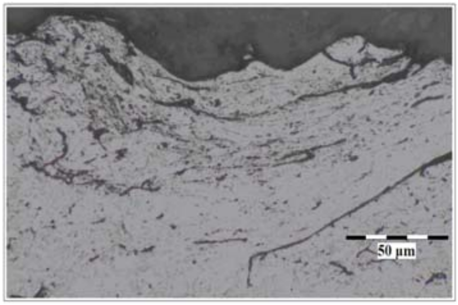


Fig.16. Cracks in a pitting site in shot peened material.

Along with the listed facts, it is possible to also add monitoring of the influence of Hertz stress on pitting size, Fig.14. As shown in the figure, pitting size grows exponentially with the applied stress in both types of material. It is also evident that for material without shot peening, the pitting growth is much greater than for the shot peened material. This fact is also in accordance with the observation that the resistance of nitrided material after shot peening has a greater lifetime than material that was plasma nitrided on the original non-shot peened surface. Furthermore, this measurement supports our previous analysis of the mechanism of pitting formation on shot peened and non-shot peened materials.

CONCLUSIONS

1. Plasma nitriding applied to a classical type of sintered material Astaloy CrM with 0.3% C showed that this technology is no significant improvement when compared with other surface treatments including classical gas nitriding.
2. The application of surface shot peening before plasma nitriding for this material has proved to be very beneficial: an increase in fatigue strength at $50 \cdot 10^6$ cycles from 1200 to 2400 MPa, which represents an increase of 100%.
3. Metallographic microscopic analysis of processes taking place during contact fatigue showed that the failure of the surface layer by pitting formation is fundamentally different from the mechanism in material which was or was not shot peened before nitriding. The explanation of this mechanism is also an explanation of the lifetime increase for material that was shot peened before nitriding.
4. Metallographic microscopic analysis also demonstrated the mechanism of pitting formation. The cracks were formed as a result of stress concentration in pores located below the material surface at depths of maximum Hertz stress peaks in specimens without shot peening. For specimens which have been shot peened, there were cracks formed on the surface and subsequently they propagated at an angle of about 30°.
5. Depending on the Hertz stress, the size of the pitting (expressed by pit area) was exponentially increasing. For specimens not shot peened before nitriding, the area of pitting was considerably higher. Measurements by profilograph confirmed this, and also that the depth of pitting in shot peened materials was smaller than in the material which had not been shot peened.

REFERENCES

[1] Rodziňák, D. et al: Strojárstvo / Engineering, 2010, no. 12, p. 68
[2] Straton, PF., Sproge, L.: Heat Treatment of Metals, vol. 31, 2004, no. 3, p. 65
[3] Bengtsson, S., Marcu, T., Klekovkin, A. In: Proceedings of the 2008 World Congress

on Powder Metallurgy & Particulate Materials. Washington, USA, Part 6, 2008, p. 1
[4] Jandeska, W., Hoffman Slattery, GR., Hanejko, F., Rawlings, A., Murphy, T. In: Advances in Powder Metallurgy and Particulate Materials. Part 10. Chicago : MPIF, 2004, p. 35
[5] Kremel, S., Danninger, H., Altena, H., Yu,Y.: Powder Metallurgy Progress, vol. 4, 2004, no. 3, p. 119
[6] Rodziňák, D., Semrád, K., Zahradníček, R., Jakubéczyová, D., Hvizdoš, P.: Powder Metallurgy Progress, vol. 9, 2009, no. 4, p. 205
[7] Rodziňák, D., Zahradníček, R., Hvizdoš, P., Jakubéczyová, D.: Acta Metallurgica Slovaca, vol. 16, 2010, no. 1, p. 12
[8] Zhong-yu, P., Bin-Shi, X., Hai-dou, W., Chun-Huan, P: Surface and Coating Technology, vol. 204, 2010, p. 1405
[9] Janitzky, T., Sauermann, I., Beiss, P. In: Proc. Euro PM 2008. Mannheim, Vol. 3. Shrewsbury : EPMA, 2008, p. 243
[10] Sauermann, I., Janitzky, T., Beiss, P. In: Adv. Powder Metall. & Particulate Mat. 2008. Proc. CD. Part 10. Princeton : MPIF, 2008, p. 21
[11] Yoshida, A., Ohue, Y., Seki, M. In: Proc. of DETC.03 ASME 2003 Design Engineering Technical Conferences and Computers and Information in Engineering Conference. Chicago, Illinois, USA, September 2-6, 2003, p. 1
[12] Rodziňák, D., Čerňan, J., Semrád, K., Zahradníček, R. In: Inter. Conference Failures of Rolling Bearings (Poruchy valivých ložísk). Bratislava : 2010, p. 71
[13] Lipp, K., Sonsino, CM., Pohl, D., Hock, S. In: Proceedings of the 1998 PM World Congress. Granada, Spain. Vol. 3. Shrewsbury : EPMA, 1998, p. 143
[14] Alzati, L., Engström, U., Rivolta, B., Tavasci, A.: Höganäs AB company technical papers, Höganäs AB, Sweden. Presented at PM 2008 Congress in Washington, USA, June 2008 http://www.hoganas.com/en/News-Center/Published-Articles/Wear-Resistance-of-Gas-Nitrided-Astaloy-CrL-Sintered-Steel/
[15] Bergman, A.: Höganäs AB company technical papers, Höganäs AB, Sweden. In: PM^2 TEC 2001 in New Orleans, USA http://www.hoganas.com/en/News-Center/Published-Articles/Influence-of-Density-on-PM-Steel-Fatigue-Crack-Initiation-and-Propagation/