PITTING WEAR MECHANISM OF NITRIDED 30CrMoV6 STEEL SAMPLES

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Abstract

Nitrriding is a one of the processes which is used to increase material surface hardness. Nitrogen supersaturated layer on the material surface significantly decreases its friction coefficient. With decreasing friction coefficient an improvement of material wear resistance should be expected. It was proved in a numerous research works on sliding wear of nitrided layer. The changes of the microstructure in the surface of the nitrided material resulted in increasing its fatigue resistance, mostly due to residual compressive stresses introduced into the material surface during nitriding process. In this research the mechanism of one specific case of fatigue wear is described. The performed studies allowed to observe two different mechanisms of pitting wear initiation at the nitrided layer. One of them began with the formation of the pervious fatigue cracking at the material surface and in the case of second one the pitting wear was initiated at the subsurface area.

Keywords: Nitriding, surface layer, pitting wear, cracking, steel

1. INTRODUCTION

Increasing role of the nitriding process in industry in a significant way influenced the scientific community. The numerous variants of this process, the influence of the treatment parameters, and the influence of nitrided layer microstructure was investigated [1- 7]. The nitriding process changes the chemical composition of the surface and subsurface of nitrided material. Diffusion of the nitrogen to the material in the case of the steel allows obtain supersaturated solid solution of the iron. Continuous increase of the nitrogen concentration during the process results in precipitations of nitrogen compound. In the case of alloyed steel at the nitriding layer we should expect formation of the Al, Ti, V, Nb nitrides [1]. With the increase of the time and temperature of the nitriding process the depth of nitrided layer increases. Moreover, nitriding process results in increasing the surface hardness of treated material. All those phenomena affect the tribological properties of the material. Nitrogen supersaturation of the iron solid solution reduces the friction coefficient of the material surface [2,3]. Decrease of the friction coefficient in a significant way reduces the mass loss of the material in the case of sliding wear. The presence of the compressive stresses at the material surface resulting from nitriding process, reduce the tendency to material cracking during processes involving friction [4]. Also the time of the nitriding process reduce the wear ratio of the steel samples. With the increase of the time the layer of the nitrides compounds such as γ‘ and ε is formed. The presence of hard particles at the subsurface layer also increases the wear resistance of the material [5,6]. Fatigue cracking in the case of nitrided samples is mostly initiated at the subsurface area. Hindered cracking propagation at the nitrided layer results from the compressive state of stress present in that layer [1]. Also, the fretting resistance is increased by the nitriding process due to the hinder formation of the adhesive bounds at the nitrogen supersaturated layer [5]. In the case of the pitting wear the improvement of material properties is about five times greater as compared to the non-coated materials [2]. The pitting can be initiated at the three different parts of the sample. In the first case, it can be initiated at the material surface. In the second case, it can be formed at the subsurface area. Moreover, it can also be initiated at the layer - bulk material interphase area. Formation of the pitting cracks at the material surface is related to the high differences in the mechanical properties of different parts of the material surface. At the subsurface area, the pitting cracking can be initiated at the Hertz maximum shear stress level. Interphase region between the layer and bulk material can be the place of the pitting initiations when these two areas have significantly different mechanical properties [7-11]. It is important to define the mechanism and place of the pitting wear formation, because the area of the cracking formation and the further propagation of the pitting wear can affect the material wear resistance. The changes in the microstructure of the material can also change the intensity of cracking propagation. Due to that the main aim of the present study is a description of the pitting wear mechanism in the nitrided steel samples.

2. MATERIALS FOR THE INVESTIGATION

Materials for the research were three 30CrMoV9 steel samples. Before the nitriding, steel was quenched and tempered. Plasma nitriding process was used for thermochemical treatment of heat treated material. After the heat and chemical treatments the samples were tested on T-03 tribological tester. Traditionally the T-03 tester is equipped friction node with four steel balls which are moving around the metal track. For the purpose of the investigations, the one of the balls was replaced by the chamfered cylindrical sample. The schematic representation of the traditional and modified friction node is presented in **Figure 1**. This modification allowed to more precisely control the loads and also to facilitate further metallographic analysis. Pitting wear resistance test was performed at the room temperature, under the load of 3923 N. The tests were performed with a lubrication, with a mineral oil without additions of lubricate. The sample velocity was 1450 ± 50 rotations per minute. The test for each sample was performed until the first pit formation at the material surface. The presence of the pit was determined by the analysis of vibration amplitude in the friction couple. The formation of the first pit resulted in the high increase of the vibration amplitude and stopped the test. As a counter sample, the 100Cr6 hardened steel was used. For each tested samples new set of counter samples has been applied.

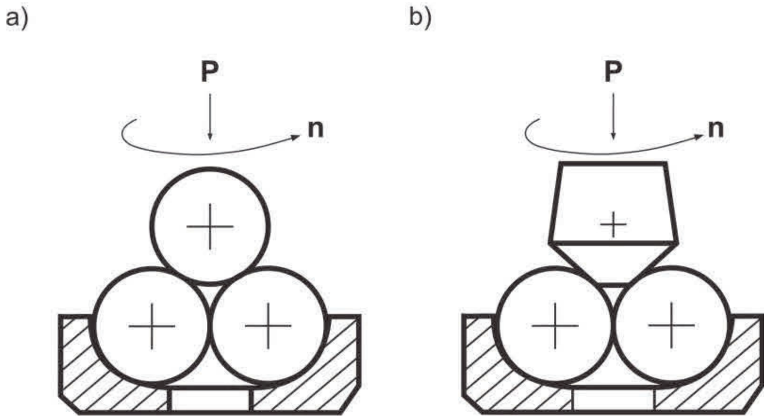

Figure 1 Friction couple in T-03 tribological tester a) original friction couple, b) friction couple after modification

After tribological tests samples surfaces were investigated on SEM HITACHI SU-70 scanning electron microscope. The pits and friction areas were observed. After surface investigations, the samples were included and grinded in the perpendicular plane to the sample axis. Further microscopic observations were performed on Axiovert200MAT light microscope and using SEM. The samples were etched with 2 % nital. The micro-hardness of nitrided layer and subsurface area of the material were examined using TUKON 2500 hardness tester. The tests were performed using Knoop indenter applying the load of 0,098 N.

3. RESUTLS OF THE INVESTIGATIONS

3.1. Fracture analysis and the microstructure of the nitrided layer

The time to the first pit formation at the samples surfaces were respectively: S1: 1101 s., S2: 11045 s., and S3 16083 s. The observations of the investigated non-etched cross sections of the samples allowed to define the area of cracking formation in each sample. For S1 sample the undersurface cracks perpendicular to the material surface were observed. At the S2 sample cross section the developed pit with the secondary cracks was observed. Also, some of the cracks on the right side of the pit were probably initiated at the material surface. For S3 sample the straight cracks initiated at the material surface were observed (**Figure 2**). Microstructures of the nitrided layers for each sample are presented in **Figure 3**. The microstructure of tested materials consisted of a tempered martensite. In the case of S1 sample, the nitrided layer was characterized by the presence of the nitrogen compound at the former austenite grain boundaries (**Figure 3a**). For the samples S2 and S3 the presence of the nitrogen compound layer (white layer) at the material surface was observed. Moreover, the presence of the grain boundary precipitations of nitrides was observed for this sample (**Figure 3b, c**). The changes of the nitrided layer morphology are probably related to the change of the nitriding potential for the samples S2 and S3, which resulted in the formation of the compound layer at the material surface [1,3]. The small changes can be explained by the inhomogeneous distribution of the compounds at the materials surface and very small thickness of the white layer.

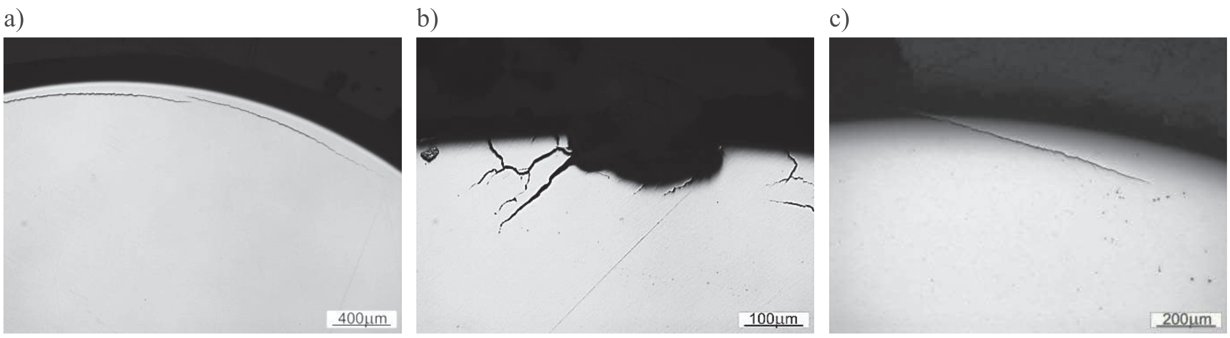

Figure 2 Surface area at the samples cross-sections: a) sample S1, b) sample S2, c) sample S3

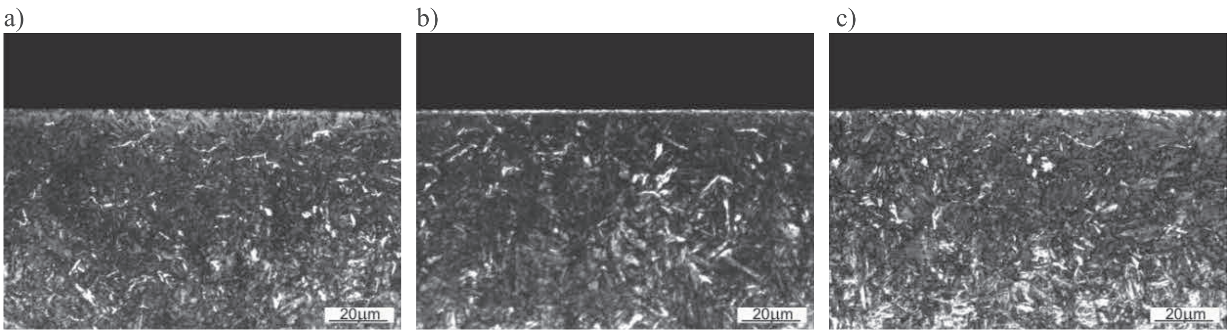

Figure 3 The microstructures of nitrided layers in the investigated samples: a) sample S1, b) sample S2, c) sample S3

3.2. The analysis and the microstructure of the nitrided layer and wear area

Scanning Electron Microscopy investigations allowed define the pits shape and the character of wear mechanism in the wear area. In the case of S1 sample, the elongated shape of the pit was observed. Fatigue cracking in the sample caused delamination of the layer. Secondary cracks, perpendicular to the pit edges, were also observed (**Figure 4**). The analysis of the pit for S2 sample allowed observation of more rounded shape of the pit. The secondary cracks initiated at the pit surface were observed. The cracks parallel to the pit edge were also visible (**Figure 5a**). Wear mechanism for sample S3 was similar as in the case of sample S2. The pit was initiated probably at the material surface. Secondary cracks, parallel to the pit edge, were also observed as in the case of sample S2 (**Figure 5b**).

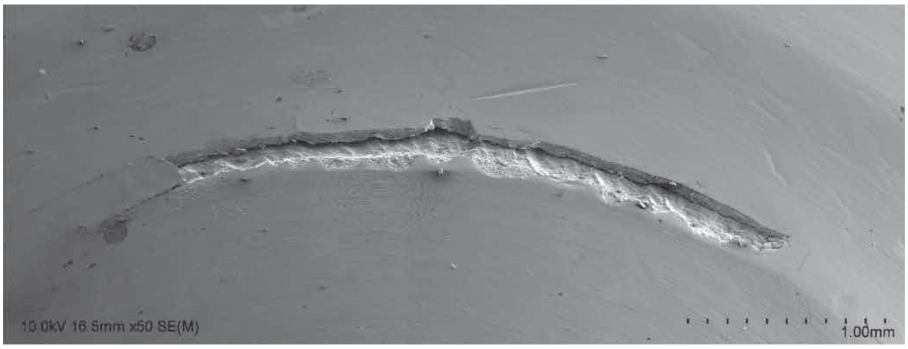

Figure 4 SEM image of wear area observed on sample S1

a) b)

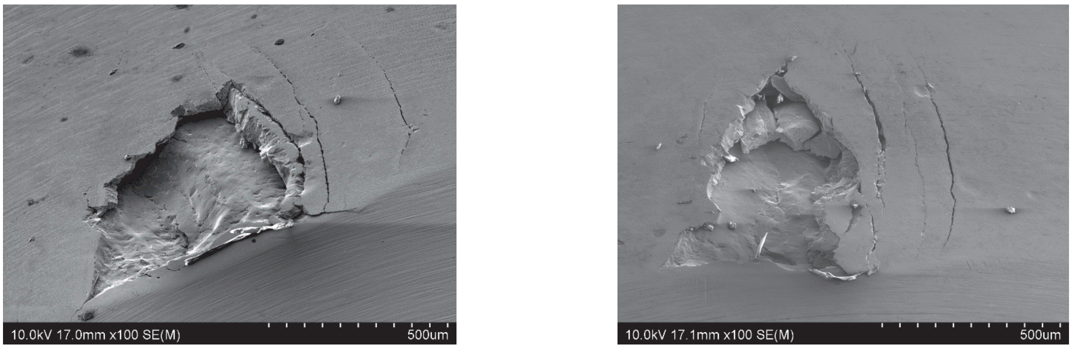

Figure 5 SEM images of wear area observed on the investigated samples: a) sample S2, b) sample S3

The observations of etched cross-sections of the samples using SEM allowed examine subsurface microstructure. For each sample the microstructure of the subsurface layer consisted of tempered martensite. Nitrocarbides precipitations at martensite needles and former austenite grain boundaries were observed (**Figure 6**). There are no clear correlation between subsurface microstructure and cracks propagation direction. Nitrocarbides precipitations favor further cracks propagation only in the case of the favorable stress state during wear (**Figure 6**).

a) b) c)

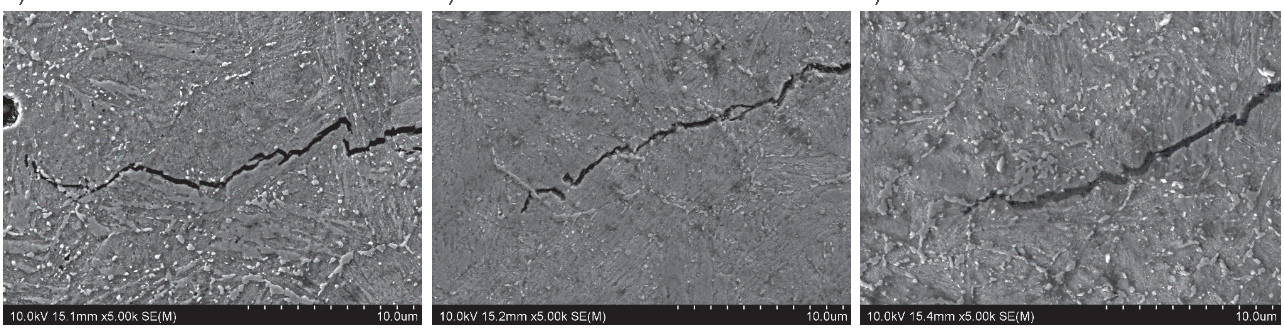

Figure 6 The microstructures of the investigated samples at the area of nitrided layer: a.b) sample S1; c,d) sample S2; e) sample S3

3.3. Microhardness measurements

Average micro-hardness for nitrided layer was respectively: sample S1: 884HK 0.01; sample S2: 940 HK 0.01, sample S3: 897 HK 0.01. The constant decrease of the material hardness with the depth of the investigation was observed. For sample S1 the hardness constantly decreased to the value measured for the bulk material. The average bulk material hardness was about 469 HK 0.01 (**Figure 7a**). Hardness of nitrided layer for sample S2 maintained at the same level as measured on the surface to about 0.18 mm sample depth. Below the nitrided layer at the distance of 0.11 mm micro-hardness was about 841 HK 0.01. Micro-hardness of the base material was about 469 HK 0.01 (F**igure 7b**). The similar as for sample S2 character of micro-hardness profile was observed for sample S3. Micro-hardness of the material decreased below nitrided layer to value about 822 HK 0.01 and after about 0.05 mm decreased steadily to the 494 HK 0.01 for the base material (**Figure 7c**).

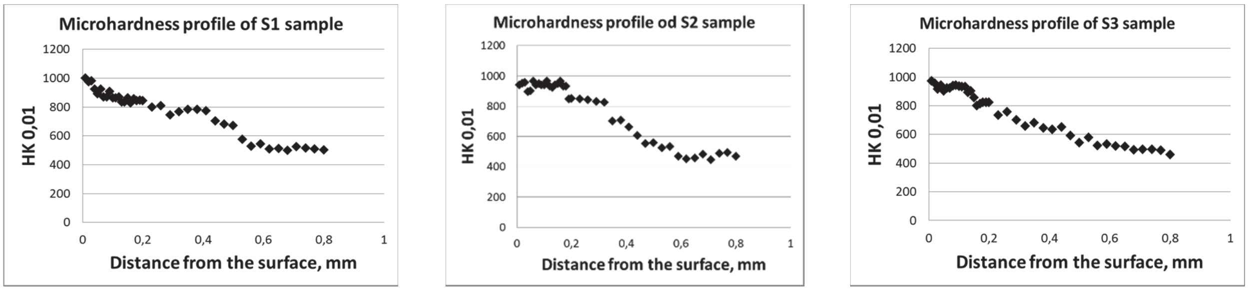

Figure 7 Microhardness profiles for the samples: a) S1; b) S2; c) S3. Knoop indenter, test load: 0,098 N

For sample S1 presented results allowed state, that pitting wear was initiated below the material surface. Fatigue cracks were initiated at the maximum shear stress area (Hertz Point) located beneath the nitrided layer. Cracking propagated at the same depth under surface, and caused delamination of surface layer. The shortest time required to failure for this sample resulted from easier cracking propagation in subsurface. The pits observed on samples S2 and S3 were initiated probably at the surface. The change of pitting wear initiation mechanism was analyzed by the changes in the pits shape and secondary cracks shape observed on the cross-sections [12]. The brittle nitrocarbides precipitations at the surface were the sources of primary fatigue cracks witch propagated perpendicular to the surface of the sample. The compression stresses in nitrided layer inhibit cracks propagation, what resulted in longer time required to pit formation. Micro-hardness profiles of the samples allow to state, that for sample S1 cracks propagated in the area of nitrided layer having lower hardness, and then propagated to the material surface. For samples S2 and S3 fatigue cracks changed direction to parallel to the surface in the area with lower hardness. Decrease of hardness with the distance from the surface probably resulted from decrease of compressive stresses [1,3,13]. Lower compressive stresses in the subsurface regions favor the crack propagation in those regions. The consequence of compressive stresses decrease was shorter time to pit formation for sample S1 and change of fatigue cracks propagation direction for samples S2 and S3. Direct correlation between the microstructure and crack propagation was not observed. The main parameter influencing cracking direction was probably the mechanical characteristic of load during the tribological tests. Former austenite grain boundary precipitations of nitrocarbides favor promote cracking only when precipitations are located at energy preferred direction.

4. CONCLUSIONS

The following conclusions can be drawn from this study:

Two different mechanisms of pitting wear initiation were observed. Fatigue cracks were initiated on and under the surface of the samples. Initiation of the cracks at the surface was caused by the presence of brittle compound layer (white layer). Continuous changes of the cyclic stresses during the tribological tests resulted in the crack initiation at mentioned point. The cracks propagated in this case perpendicular to material surface. The cracks observed under the surface of the sample were probably initiated in the area of maximum shear stresses (Hertz Point), and they propagated parallel to the surface of the sample. The cracks were initiated in the nitrided layer, in the area of lower hardness. Compressive stresses in the nitrided layer inhibit the cracks development. In the case of the cracks initiated on the surface of the sample, the presence of compressive stresses caused prolongation of time to pit formation. In the case of the cracks initiated under the surface, probably in the area of lover compressive stresses, the shortest time required to pit formation was observed. There was no direct correlation between the microstructure of nitrided layer and cracking direction. Nitrocarbides precipitations promote cracking when their locations correspond to the state of stress in the material. Beyond these cases there was no direct correlation between microstructure of nitrided layer and cracking direction.

ACKNOWLEDGEMENTS

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REFERENCES

[1] MENTHE, E., RIE, K.T., SCHULTZE, J.W., SIMSON, S., Structure and properties of plasma - nitrided stainless steel. Surface and Coatings Technology, 1995, vol. 74 - 75, pp. 412 - 416.

[2] SUBBIAH, R, RAJAVEL, R, Dry sliding wear behavior analysis of nitrided 316L grade austenitic stainless steel using gas nitriding process, Journal of Theoretical and Applied Information Technology, 2010 pp. 98 - 101

[3] KUKSENOVA, L. I., LAPTEVA, V. G., BEREZINA, E. V., GERASIMOV, S., SENATORSKI, Y. Structure and wear resistance of nitrided steel. Metal Science and Heat Treatment, 2004, vol. 46, pp. 32-35.

[4] Skakov, M. K., Rahadilov, B. K., Shceffler M. Study of nitrided steel R6M5 abrasive wear -resistance. In *40th Leeds-Lyon Symposium on Tribology and Tribochemistry Forum*. Lyon: 2013, pp. 124-130.

[5] GENEL, K., DEMIRKOL, M., CAPA ,M. Effect of ion nitriding on fatigue behavior of AISI 4140 steel. Material Science and Engineering, 2000, vol. A279, pp. 207 - 216 .

[6] KRAWCZYK, J., BAŁA, P., Optimalization of heat and thermo-chemical treatment of 50CrMoV18-30-3 steel for hot forging dies. Archives of Metallurgy and Materials, 2009, vol. 54, pp. 233 - 239.

[7] STEWART, S., AHMED, R. Rolling contact of surface coatings - a review. Wear, 2002, vol. 253., pp. 1132-1144.

[8] DATSYSHYN, O. P., PANASYUK, V. V., GLAZOV, A. Y. Modeling of fatigue contact damage formation in rolling bodies and assessment of their life time. Wear, 2011, vol. 271, no. 1-2, pp. 186-194.

[9] FAJDIGA G., GOLDEZ S., FLAS J. Numerical simulation of surface pitting due to contact loading, International Journal of Fatigue, 2001, vol. 23, pp. 599-605.

[10] GLODEŽ, S., ABERŠEK, B., FLAŠKER, J., REN, Z. Evaluation of the service life of gears in regard to surface pitting, Engineering Fracture Mechanics, 2004, vol. 71, no. 4-6, pp. 429-438.

[11] PANASYUK, O. P. D., PANASYUK, H. P. M. V. V., MARCHENKO, H. P. The crack propagation theory under rolling contact. Engineering Fracture Mechanics, 1995, vol. 52, no. 1, pp. 179-191.

[12] FAJDIGA, G., GLODEŽ, S., KRAMAR, J. Pitting formation due to surface and subsurface initiated fatigue crack growth in contact in mechanical elements. Wear, 2007, vol. 262, no. 9-10, pp. 1217-1224.

[13] CHUE, C. H., CHUNG, H. H., LIN, J. F., CHOU, C. C., The effects of strain hardened layer on pitting formation during rolling contact. Wear, 2001, vol. 249, no. 1-2, pp. 108-115.