INFLUENCE OF INCLUSIONS GEOMETRY ON THE FATIGUE PROPERTIES

STUDENY Zbynek, POKORNY Zdenek

Faculty of Military Technology, University of Defence, Kounicova 156/65, 662 10 Brno, Czech Republic, EU, zbynek.studeny@unob.cz, zdenek.pokorny@unob.cz

Abstract

The inclusions size in context fatigue properties of plasma nitriding steel was studied. The size of inclusion, such as geometry and location, has remarkable influence to fatigue properties of steel 41CrAlMo7-10. The samples were shaped by turning operations and subsequently grinded to prescribe values of a standard test bars. Next technological operation was heat-treatment, such as normalizing, quenching and tempering. Plasma nitriding technology as a diffusion technology used to formation of nitrided layers on surface of heat-treated steel. Plasma nitriding technology is effective method usage in practise especially due to increasing of surface hardness, corrosion resistance and fatigue strength. The samples were subjected to the fatigue bending rotation tests.

Keywords: Plasma nitriding technology, fatigue test, inclusions

1. INTRODUCTION

Plasma nitriding technology is a thermo chemical procedure based on diffusion of nitrogen into the surface of steels. This diffusion process is based on the solubility of saturated nitrogen in crystal lattice [1]. The saturation of nitrogen cause the formation of positive nitrides on surface and or course the formation of diffusion layer. The thicknesses of nitride layers is dependent on chemical composition of nitride formed elements [2].

Nitriding equipment is essentially a vacuum container (recipient), where is the container wall connected as the anode, while the nitrided component must by plugged in the cathode. The largest voltage drop occurs in a few millimeters from the surface of the component, this fact is appeared as light emitting of glow discharge. The electric field creates around of surface of batch glow discharge which causes the ionization of gas or mixture of gases, a.g. N_2, H_2. Positive ions are accelerated towards the batch, impacting on it with high kinetic energy. Part of the incident ions is converted into heat, heating the batch evenly. Temperature of the component in the plasma nitridation furnace, as in the other the diffusion methods, is crucial because the diffusion coefficient heavily depends on [2].

The formation of the nitride layer on surface of material can be essentially controlled allows to ensure the changes of process parameter as voltage, temperature, surface current density, pressure, composition of atmosphere and nitriding time during plasma nitridation [2].

The experiments were focused on steel 41CrAlMo7-10. The samples were classified into two groups (A10 and A30). Two series of samples were heat treated and plasma nitrided under different environment conditions in the Rübig PN 60/60 furnace. The experiment proceeded testing in fatigue three-point bending rotation tests. The fatigue tests were performed on Instron R.R.Moore L2568 machine. Methodology of testing was in accordance with Czech standard [3] and statistical evaluation of fatigue test results was in accordance with Czech standard [4]. Fatigue fractures was documented and evaluated by using REM electron microscope Tescan Vega TS 5135.

2. EXPERIMENT DESCRIPTION

The experiment work consisted of sample preparation, fatigue tests with statistical evaluations and fracture analysis. For experiment two series of samples (A10 and A30) were used. The differences between series of samples are evident in **Table 2**.

2.1 Sample preparation

Standard test bars samples were made from steel rods of diameter 25 mm. The experimental samples were turned and grinded to prescribe values of a standard test bars. Diameter of fixture portion was 7,61 mm and diameter of experimental portion was 5,2 mm. Accuracy of production was 0,01 mm.

Chemical analysis was performed by the optical emission spectrometry GDOES (Glow Discharge Optical Emission Spectrometry) on SA 2000 Leco instrument. Optical emission spectrometry is suitable for the analysis of all conductive metallic materials and is based on excitation of atoms sputtered from the surface of the sample material by the effect of glow discharge. The measured results are given in **Table 1**. Calibration of the spectrometer was performed using CKD 150A up to 189A etalons.

Table 1 Chemical composition of testing steel 41CrAlMo7-10

Element	C	Mn	Cr	Mo	V	Cu	Si	P	S
Wht %	0.31	0.38	2.25	0.21	0.28	0.05	0.25	0.007	0.009

Experimental samples were heat-treated. The parameters and procedure of heat-treated operation are given in **Table 2**. In case of plasma nitriding was an environment inverted.

Standard DIN 50190 [5] was applied in the thickness measurement of nitrided layers. The microhardness and the depth of the nitrided layer was evaluated on automated testers LM247AT LECO with AMH43 software. Test load was set at 50 g. The particular microhardness values are plotted in the graph **Fig. 1**, from which can be effectively identified the limit hardness of the layer. Counted value express the hardness of the base material increased by 50 HV [1]. In accordance with DIN 50190 standard [5] was following equation used for calculation of nitrided layer thickness X (1).

$$X = ((Y * 0,1) * 10) + 50 \qquad (1)$$

Where, X is nitrided layer thickness in mm, Y is the average microhardness number from five indentation´s patterns in HV 0.05 [kg]. Depth of the nitrided layer is practically determined by 20 indentations, the first of them starts closest to the edge of the sample. Spacing of individual indetations was set from 0.01 mm to 1 mm.

Table 2. Samples heat treatment procedure

Technology	Series of samples	Temperature [°C]	Time	Environment	U [V]	Pressure [Pa]	Pulse length [μs]
Normalizing	A10, A30	900	25 min	air	-----	-----	-----
Quenching	A10, A30	930	25 min	oil	-----	-----	-----
Tempering	A10, A30	640	40 min	oil	-----	-----	-----
Nitriding	A 10	500	10 h	8:24 (H_2:N_2) [l/h]	530	280	120
	A 30	500	10 h	24:8 (H_2:N_2) [l/h]	530	280	120

2.2 Fatigue tests

Fatigue tests were done under the same conditions in a controlled environment in case of two series of samples marked as A10 and A30 according to the CSN 42 0363 standard [3]. Fatigue tests were performed on a Instron R. R. Moore L2568 test machine. Each serie included 15 samples and 3 samples were tested on each loading level with interval 15 MPa between loading levels [3].

2.3 Fracture analysis

Fracture analysis were performed on a REM electron microscope Tescan Vega TS 5135. The analysis consisted of observation fracture and measuring an inclusions. An inclusion, such an initiation place, has a direct influence on the method and speed of propagation of fatigue cracks. Effects of inclusions to the fatigue limit were discussed mainly in works of Murakami [6]. For the value of stress intensity factor on the surface of ellipsoidal inclusion K_{Imax} in [MPa.m^{0,5}] he deduced the formula:

$$\Delta K_{\mathrm{Im}ax} \approx 0{,}65.\Delta\sigma.\sqrt{\pi.\sqrt{A_p}} \quad , \qquad (2)$$

where Δσ [MPa] is the applied stress range and A_p [m] is the area of the inclusion projected on a plane perpendicular to the loading direction [7]. Based upon ΔK value is the driving factor for the crack growth rate, there is a tendency of dependence between applied stress range and fatigue live represented by numbers of cycles to fracture machine parts [8].

3 DISCUSSION OF RESULTS

3.1 Sample preparation

The surface microhardness value was measured on the cross-sectional samples at precisely defined distances from the surface. The obtained values are presented as a funciton of distance from the surface see **Fig. 1**. All measurements were done under the same conditions, including sample preparation. Microhardness measurement was done on every sample dependent on the distance from edge. According to DIN 50190 [5] was microhardness profile of the nitride diffusion layer evaluated. The microhardness value of serie A10 has 390 HV0.05 in depth 264 μm below the surface and the microhardness value of serie A30 has 383 HV0.05 in depth 227 μm below the surface. The depth 264 μm (serie A10) and 227 μm (serie A30) expresses transition between diffusion layer and core of material.

A thinner diffusion layer in case of A30 serie beside serie A10 was caused by an inverted the environment in the plasma nitriding process. According to **Table 2** had serie A30 three times less nitrogen in plasma nitriding process than serie A10.

3.2 Fatigue tests

In accordance with CSN 42 0368 standard [4] two Wöhler`s S-N curves were created and results are given on **Fig. 2**. The curves expressed the influence of the plasma nitriding on fatigue life. Wöhler`s curves show dependence between Stress amplitude σ_a in MPa and Number of cycles displayed in logarithmic coordinate.

The Wöhler`s S-N curves were obtained after 15 tested samples for both of series of samples A10 and A30. The fatigue life value was for serie of samples A10 caltulated at level 665 MPa and for serie of samples A30 at level 660 MPa. It can be concluded according to the principle of the fatigue life values calculation in accordance with Czech standard [4] the values of fatigue life of both series of samples A10 and A30 are equal. Not less important on **Fig. 2** is area of fatigue strength for finite life. It is inclination part of Wöhler`s S-N curves. Area of fatigue strength for finite life of series A10 and A30 is equidistant. The distance between inclination parts of Wöhler`s curves is approximately 15 MPa of series A10 and A30.

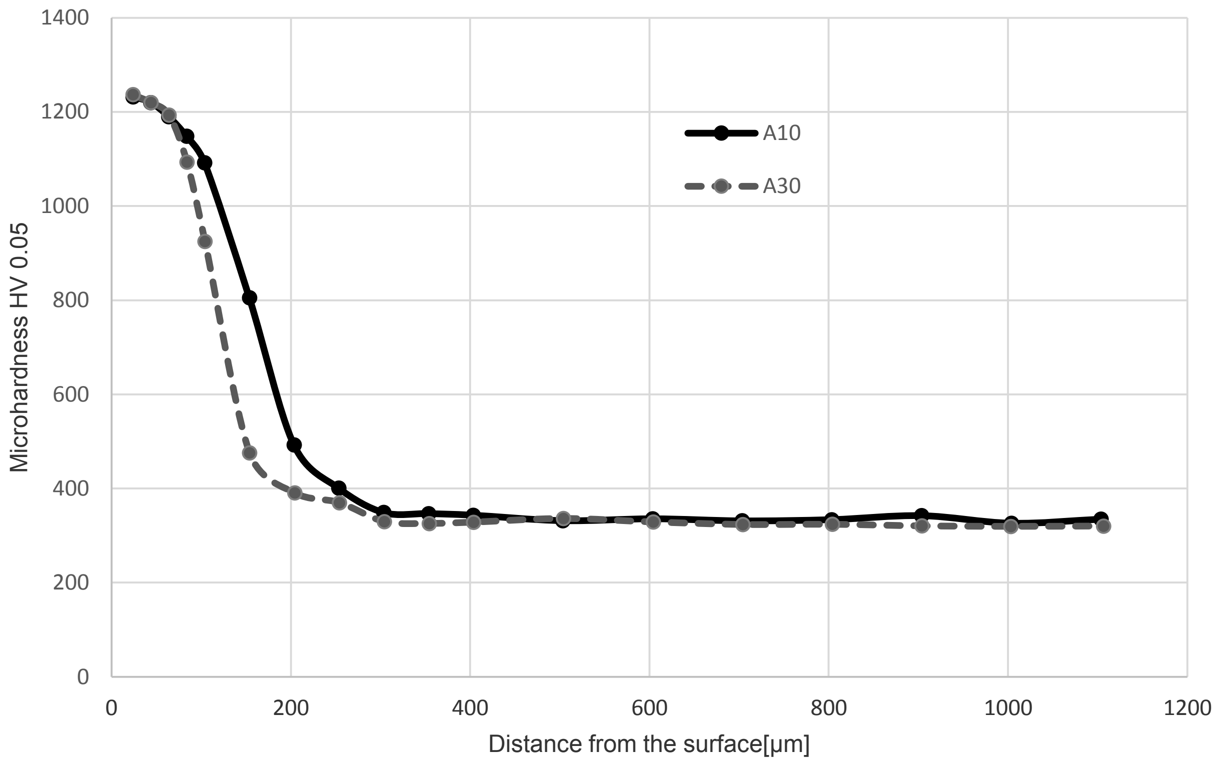

Fig. 1 Avarage microhardness trend HV 0.05 serie of samples A10 and A30

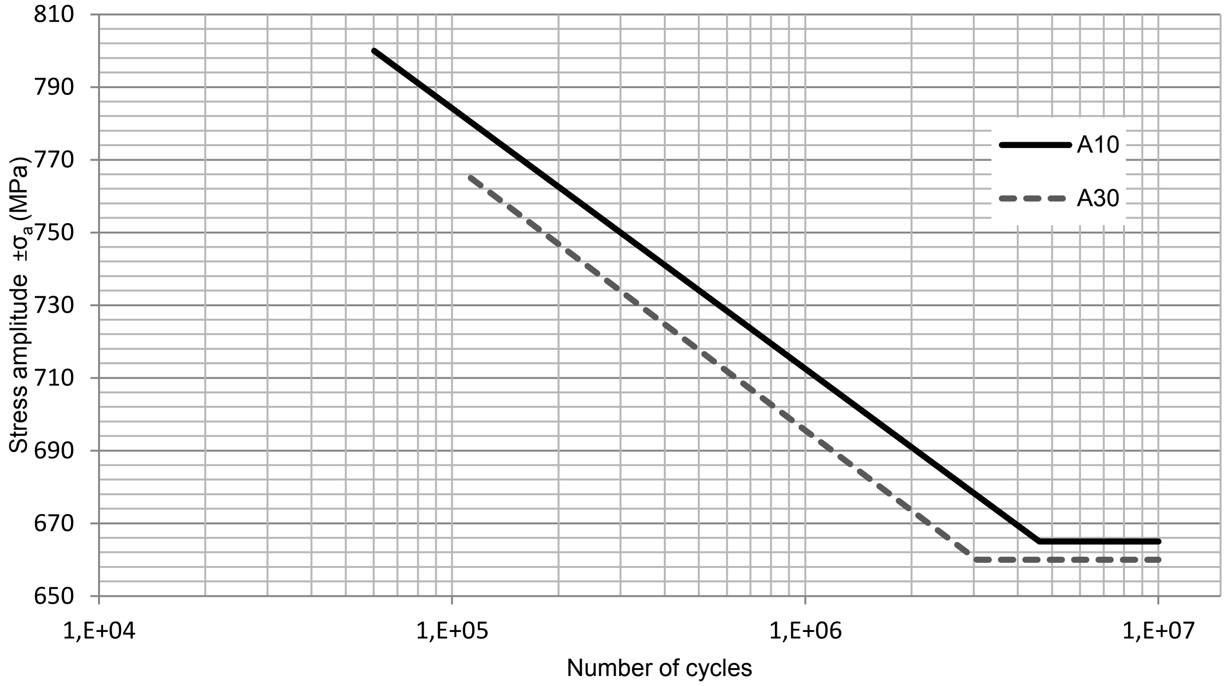

Fig. 2 Wöhler`s S-N curves serie of samples A10 and A30

3.3 Fracture analysis

Measuring and documenting of inclusions geometry was performed by REM electron microscope Tescan Vega TS 5135. Sample of inclusion series A10 see **Fig. 3** and series A30 see **Fig. 4** are displayed. Inclusions involves different geometry as a cubic or spherical. For calculation progress of stress intensity factor ΔK see **Fig. 5** series of samples A10 and A30 is necessary to know value of inclusions area.

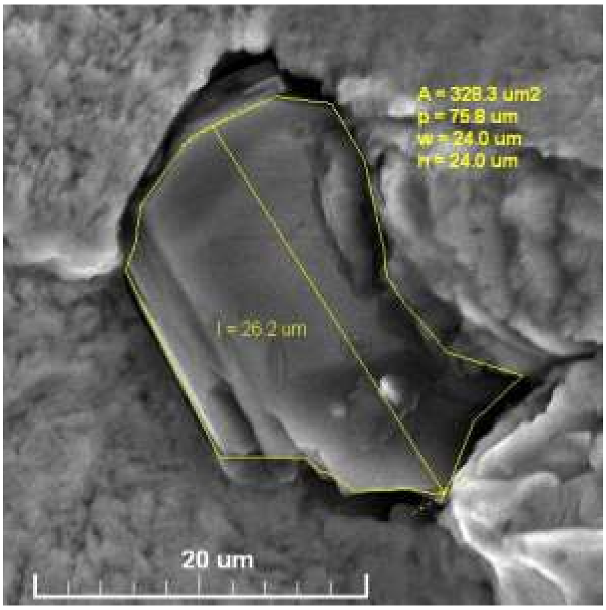

Fig. 3 Inclusion serie A10, magnifiacation 4000x SE

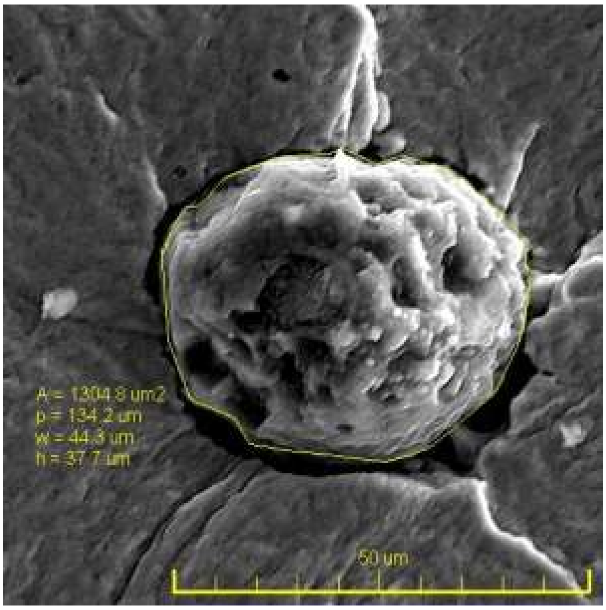

Fig. 4 Inclusion serie A30, magnifiacation 2000x SE

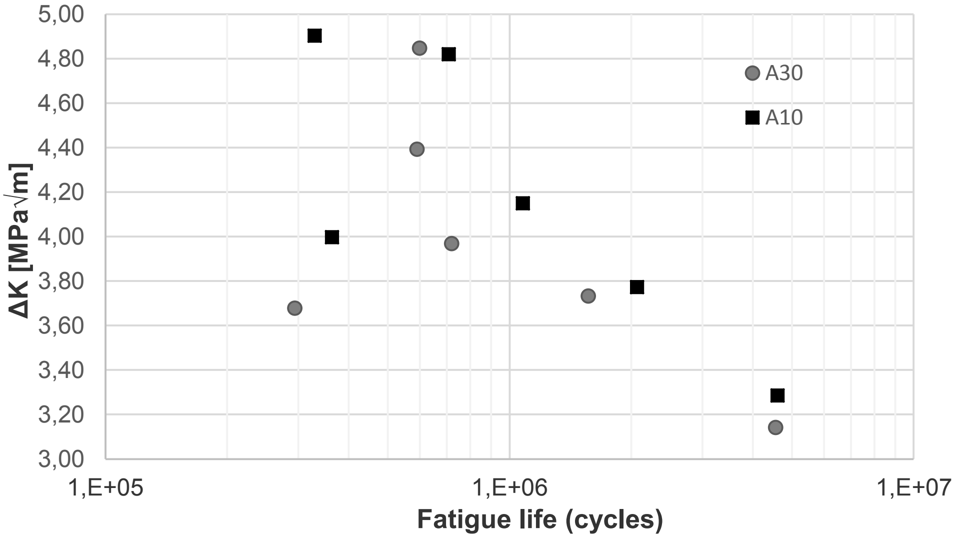

Fig. 5 Progress of stress intensity factor ΔK series of samples A10 and A30

Values of stress intensity factor ΔK series of samples A10 and A30 were calculated according to the equation (2). The trend of dependence stress intensity factor ΔK is decreasing on increasing number of cycles see **Fig. 5**. It follows dependence decreasing area of the initiation inclusions with decreasing stress on increasing number of cycles to fracture. Stress intensity factor ΔK trend is in accordance with Wöhler`s S-N curves trend in **Fig. 2**.

CONCLUSION

The paper was focused on evaluation of fatigue of plasma nitrided samples. The samples were divided into two groups based on environment plasma nitriding conditions marked as A10 and A30. Each group had 15 samples. Plasma nitriding process duration for series of samples A10 and A30 was 10 hours.

The value of microhardness trend of series A10 and A30 were evaluated on automated testers LM247AT LECO with AMH43 software. The depth of diffusion layers equals 264 μm in case of series A10 and 227 μm in case of series A30. The fatigue tests series A10 and A30 were performed on Instron R.R.Moore L2568 machine. Resulting Wöhler`s curves were equidistant. Values of fatigue life was for serie A10 determined at level 665 MPa and for serie A30 was value of fatigue life determined at level 660 MPa. Using a revers environment in plasma nitriding proces in case of serie A30, fatigue life is almost identical as in case of serie A10. In ratio $1N_2 : 3H_2$ environment serie A30 were used three times less nitrogen then in case of serie A10.

Fracture analysis were performed by REM electron microscope Tescan Vega TS 5135. Values of inclusions cross section were determined. Based on the cross section inclusion and Murakami`s formula were values of stress intensity factor ΔK series of samples A10 and A30 determined. Dependence ΔK - stress intensity factor and Fatigue life in cycles show in case of:

- gas ratio $3H_2 : 1N_2$ (serie A30) low content of nitrogen caused decreasing of critical geometry of initiation inclusion. The geometry of inclusion was observed smaller the in case of gas ratio $1H_2 : 3N_2$ (serie A10).

ACKNOWLEDGEMENTS

The paper was prepared with the support of the Project for the Development of the Organization of the Dep. of Mechanical Engineering, UoD "Promoting Research, Science and Inovation in the Field of Engineering".

REFERENCES

[1] STUDENÝ Z., KUSMIČ D. Influence of Inclusions Size On The Nitrided Components Fatigue Life. In METAL 2014: 23rd International Conference on Metallurgy and Materials. Ostrava: TANGER, 2014, pp. 875-880.

[2] HRUBÝ V., LIPTÁK P., POKORNÝ Z., Plazma nitriding of cavities, Rzeszów, 2013. ISBN 978-83-63666-93-4

[3] ČSN 42 0363 Metal testing. Fatigue testing of metals. Methodology of testing.

[4] ČSN 42 0368 Metal testing. Fatigue testing of metals. Statistical evaluation of fatigue test results of metals.

[5] DIN 50190 – part 3: Hardness depth of heat-treated parts; determination of the effective depth of hardening after nitriding.

[6] MURAKAMI Y., Metal Fatigue, Effect of small defects and nonmetallic inclusions. Elsevier publ., 2002, Oxford, UK.

[7] LAMBRIGHS K., at col. Influence of non-metallic inclusions on the fatigue properties of heavily cold drawn steel wires. Procedia Engineering 2, 2010, p. 173-181.

[8] STUDENÝ Z. Analysis of the influence of initiating inclusions on fatigue life in plasma nitrided steels. Manufacturing Technology, 2015, vol. 15, no. 1, p. 99-105. ISSN 1213-2489.