

## INFLUENCE OF INCLUSIONS SIZE ON THE NITRIDED COMPONENTS FATIGUE LIFE

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#### Abstract

The paper is focused on the fatigue strength in material research. The aim was evaluation of influence of plasma nitriding technology to fatigue strength. Plasma nitriding is a thermo chemical procedure based on diffusion of nascent nitrogen into the surface of steels. This diffusion process is based on the solubility of nitrogen in metal matrix and nitrides creation.

Especially at the point of fatigue fracture initiation there was in more detail the mechanism of fatigue failure examined. The inclusion area is a place where usually the fatigue fractures arise. The inclusion is subjected to a thorough examination of the impact in terms of the size of inclusions on the tension amplitude dependence and an analysis of the inclusions chemical composition.

Based on performed fatigue tests and won results can be submitted, that the plasma nitriding technology has a positive influence to fatigue strength of machine parts. The fatigue tests performed using the Instron R. R. Moore L2568 device established an increase of fatigue life more than 60 %. The initial amplitude of tension 416 MPa (basic material - heat-treated) was increased after plasma nitriding procedure to 665 MPa. Plasma nitriding treatment was realized under reverse nitriding atmosphere conditions  $1H_2:3N_2$  (I/h), to promote the dominant formation of Fe<sub>4</sub>N phase in the compound layer and so-called white layer. Experimental results were summarized and the Wöhler's (S-N) curves were created.

Keywords: Fatigue live, inclusion, plasma nitriding

## 1. INTRODUCTION

The studies of fatigue degradation parts of machines last over two centuries. It has brought a very wide file of information about material characteristic under precisely defined conditions [1-3]. Mechanical properties change i.e. mainly fatigue live of materials is usually describe by well known Wöhler's (S-N) curve. This curve describes dependence of number of cycles to failure N<sub>f</sub> on stress amplitude dependence  $\sigma_a$  in MPa. Therefore, since the number of cycles to the failure is a very large N<sub>f</sub> is usually indicates, in logarithmic coordinates. Nowadays the dissemination stage fatigue fractures are well known. As first there are some changes of mechanical characteristic. In second stage, we can see a crack nucleation and dissemination of short cracks. Finally in last stage there is dissemination of long crack finishing by static fracture. Cross-sectional weakening and still operating stress amplitude has an effect on static fracture of components [4-6]. In second stage we can see stress nucleation in the surface layer of the material. Stress nucleation usually occurs at the inhomogeneity. It could be like the inclusion or cavity from which is radial striation spreading. This radial striation has on cross sectional view several times larger area than the area of stress nucleation and typically take the shape of an ellipse. Just for their shape are called as "fish eye".

In the paper the experiments are focused on steel CSN 41 5340 (corresponds 41CrAlMo7-10 or 1.8509) samples. The samples have been heat treatment and follow-up plasma nitrided in the Rübig PN 60/60 equipment. Subsequently they have been subjected to the fatigue three-point bending rotation tests using the Instron R.R.Moore L2568 device. The documentation of fatigue cracks pictures have been investigated using the REM electron microscope Tescan Vega TS 5135. There were measured the contents of inclusions areas.



The REM device is coupled to EDS device. Thanks this combination was possible the chemical composition evaluation of the fracture interest area.

## 2. SAMPLE PREPARATION

The main idea of this work was fatigue limit shift calculation. The rods samples were made of diameter 25mm turning with added. The added material was after heat treatment grinded away. For chemical composition checking was the GDOES/Bulk method used (SA 2000 Leco device). **Table 1** shows the chemical composition of the basic material.

Before the plasma nitriding process, for nitriding parameters see **Table 2**, were the steel specimens heat treated under following conditions: - normalized: 900 °C / 25' in air, than quenching: 930 °C/ 25' in oil and tempering: 640 °C/ 40' in oil. After heat treatment was a reference sample cross-sectional cutted by a metallographic saw, grinded and polished. The microstructure after etching was visible and using the optical microscope Olympus SZX9 was documented and evaluated. Martensitic microstructure was confirmed.

As final surface treatment step was the diffusional nitride layer prepared, by plasma nitriding technology [7]. The nitride layer was documented and characterized by microhardness profile testing on cross-sectional specimen, in direction form surface to the core. The nitride layer depth was determined according to the DIN 50190 standard. According to the DIN 50190 standard the diffusion layer-core border is by the core hardness HV + 50 units determined. The microhardness testing was using the LECO LM 247at device evaluated. The core material microhardness was by the value of 390 HV<sub>0.05</sub> determined in the depth of 264  $\mu$ m. The evaluation was on 5 samples of A1 set of samples performed. For the calculated microhardness profile see **Fig. 1**.

Element	С	Mn	Cr	Мо	V	Cu	Si	Р	S
weight %	0,31	0,38	2,25	0,21	0,28	0,05	0,25	0,007	0,009

**Table 1** Chemical composition of the CSN 41 5340 steel



Distance from the surface (µm)

Fig. 1 Average calculated microhardness profile HV 0.05 A1 set of samples

Table :	2	Plasma	nitriding	conditions
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Steel /sample	Temperature	Duration	U	р	Pulse length	Gas flow (l/h)	
	(°C)	(h)	(V)	(Pa)	(µs)	H <sub>2</sub>	N <sub>2</sub>
15 340 / A1	500	10	530	280	120	8	24



# 3. EXPERIMENTAL

## 3.1 Fatigue tests

High-cycle fatigue tests were using the Instrom R.R.Moore L2568 device performed. The device allows to invoke symmetrically alternating rotor displacement cycle by three-point bending station, asymmetry parameter R = -1. The experiment starts by testing of basic material (A0.1 set of samples), followed by testing of plasma nitride steel samples (A1 set of samples). Every set of samples consists of 15 samples. The test rods were stressed by set loading levels of each of two to three samples for each level. The values of account of cycles to fracture resulting into the Wöhler's (S-N) curve of every set of samples was constructed by average values of particular measured cycles to the fracture according to [3, 4, 8, 9]. The results were summarized and the Wöhler's (S-N) curves were constructed (see **Fig. 4**), discussed in following chapter.

## 3.2 Fracture analysis

The cracks initiation area was by the REM evaluated. The fish eye area was evaluated, which is by shape of ellipse characterised, see **Fig. 2**, especially the initiation site i.e. the inclusion, see **Fig. 3**. The device was used for measuring these ellipses and calculates the area or perimeter. After fish eye area measuring was the size of initiating inclusion measured. In the case of missed inclusion was the inclusion depression measured, representing the cross-sectional area. For the loading level dependence on the surface inclusion see **Fig. 5**. On the same loading levels were the numerical values of measured areas averaged.



Fig. 2 A1-11 sample, fish eye 120x SE

Fig. 3 A1-11sample, inclusion 4000x SE

# 4. DISCUSSION

## 4.1 Fatigue tests

High-cycle fatigue tests were at first on the A0.1 set of rods samples performed. The resulting value of the fatigue limit on the horizontal part of S-N curve was on the value of 416 MPa determined. The fatigue limit for the second set of rods (marked as A1) was determined on the value of 665 MPa. The Wöhler's (S-N) curves of both of sets of the CSN 41 5340 steel rods are summarized in the **Fig. 4**, expressed as dependence of number of cycles to the failure N<sub>f</sub> indicated in logarithmic coordinates, to the stress amplitude  $\sigma_a$  in MPa. See



the increased fatigue limit up to 60 % for the plasma nitrided rod A1 set of rods. The nitride layer depth of plasma nitrided steels reached the value of 264  $\mu$ m. The performed high-cycle fatigue tests demonstrated the suitability of plasma nitriding technology for fatigue strength increase.

## 4.2 Fatigue cracking nucleation

The inclusion localized beneath the nitride layer causes fatigue cracking nucleation. From the initiation place are the radial striations an elliptical area of fine structure forming, called as "fish eye". The edge of the fish eye structure extends to the half of nitride layer depth. The REM device was used for size of initiating inclusion measuring. According to **Fig. 5**, it is evident that the size of initiating inclusions is rather increasing trend with increasing load amplitude.



Fig. 4 The CSN 41 5340 steel Wöhler's curves, heat treated (A0.1) and plasma nitrided (A1)

For understanding of inclusion characteristics, which is initiating the fatigue crack nucleation, was the chemical analysis on selected inclusion performed (sample A1-11). Chemical analysis was performed using the EDS device, see **Fig. 6a - 6d and 7a**, **7b**. Three methods were used for the chemical analysis. At first was the local chemical composition evaluation performed - in three locations. The majority element is aluminium, with average value of 78 weight %. The second type of chemical analysis was element mapping; see **Fig. 6a and 6d**. The method of areas mapping is used for elements identification on the surface. Chemical analysis is performed point by point of the whole surface sample. The results of measuring the amount of the selected elements are presented in Figure on the same scale as the size of the sample area. See especially **Fig. 6b**, for the majority of Al for inclusion composition. Elements of surrounding material in an amount corresponding to the steel see **Fig. 6c and 6d**.







Fig. 6a Sample A1-11, Fig. 6b location of Al



Fig. 6c location of Mn, Fig. 6d location of Cr





The last third method of inclusion's chemical analysis was the line scanning for selected elements across the inclusion, see **Fig. 7a**. The line scanning method is based on quantity measurement of elements like the

Fig. 5 The inclusion size on load amplitude dependence



Fig. 7a The A1-11 sample line scan

**Fig. 7b** The course of the elements of interest over the

inclusion

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previous method. The main difference is in the shape of the measured surface. Chemical analysis is performed only in the line. The thickness of this line is defined by a point. Length is given by the measured areas for example inclusions size. Thanks the Al in the course of the graph (**Fig. 7b**) is clearly visible the size of the inclusion. Size of inclusions is given by the rapid increase in its main element (Al) on the one hand and on the other hand by the sudden decrease. The slight decrease in the amount of aluminum are determined the heterogeneity of the inclusions. It is possible to measure the content of majority element and based on previous knowledge of the chemical composition, the length of inclusion too.

## CONCLUSION

Based on measurements and won graphs, see **Fig. 4**, it is clear, that the plasma nitriding technology has a positive influence on the fatigue live of machine parts. As mentioned in chapter 3.1, increased fatigue life up to 60 % was achieved, from the initial value of 416 MPa (heat treated only) to the 665 MPa (plasma nitrided). The fatigue tests were performed using the Instrom R.R.Moore L2568 device.

As next was the role of inclusion investigated, as a fatigue cracks initiator. After 15 samples measuring was evident, the growing dependence of load amplitude to increasing size of initiating inclusions. For investigation and documentation of inclusions was the REM device used. By coupled EDS to the REM device was the chemical composition of inclusion investigated. The initial inclusion consists of AI of average value of 78 weight % (see **Fig. 6a - 6d, 7a, 7b**).

## 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] Lukáš, P., Kunz, L. Foreword Fatigue 2010. Procedia Engineering 2, 2010, p. 1
- [2] Mughrabi, H. Fatigue, an everlasting materials problem still en vogue. Procedia Engineering 2, 2010, p. 3-26
- [3] Studený, Z., Hrubý, V., Horák, V. Fatigue tests of nitrided rods. *Hutnické listy*, 2012, sv. 65, č. 5, s. 30-34.ISSN 0018-8069.
- [4] Kuffova, M., Bella, V. Fatigue resistance of magnesium alloy AZ 91D. Metallic materials, 2009, Vol. 47, Issue 6, p. 415-420
- [5] Giancane, S., Nobile, R., Panella, F.W., Dattoma, V. Fatigue life prediction of notched components based on a new nonlinear Continuum Damage Mechanics model. Procedia Engineering 2, 2010, p. 1317-1325
- [6] Sugimoto, K., Fiji, D., Yoshikawa, N. Fatigue strength of newly developed high-strength low alloy TRIP-aided steels with good hardenability, Procedia Engineering 2, 2010, p. 359-362
- [7] Holemář, A., Hrubý, V. Plasma nitriding in practice, SNTL, Prague 1989, ISBN 80-03-00001-7
- [8] ČSN 42 0363 Methods of fatigue testing of metals.
- [9] CSN 42 0368 Fatigue testing of metals. Statistical evaluation of fatigue test results of metals.