

FATIGUE FRACTURE OF NITRIDED AND CARBONITRIDED LAYERS

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The phenomenon of fatigue fracture of nitrided and carbonitrided layers is described in the paper. Fatigue behaviour is described by analysing properties of the substrate and surface layer. The effect of microhardness of the substrate on fatigue properties of nitrided layers is obtained. Smith's diagrams for carbonitrided layers are presented. A graphical pattern for prediction of the place of failure initiation is suggested. Future directions of the examination with tribology and corrosion effects taken into account are marked in the paper.

Key words, fatigue fracture, nitriding, carbonitriding, hardness, fracture pattern

1. Introduction

The level of applied technology is the decisive factor for product competitiveness in the world market, but definitely not the only one. How competitive a product is depends on the entire process of manufacturing, service and recycling possibilities.

Among the most critical phases in this cycle are:

- the phase of calculation and design
- the phase of attaining material existence through various technological processes
- the service phase.

Added to the above is the final phase connected with utilization of used and worn products for new manufacturing processes.

All the above mentioned phases form the product's "lifetime". The functionality of surface layers means the ability to fulfill requirements placed on the product in given service conditions. Utilization of products, which, in the majority of cases are machine components or assemblies, takes place in conditions where the product may be exposed to mechanical loading, thermal and/or chemical environment, and other hazards.

Among mechanical loadings, the most significant are those which vary with time, i.e. fatigue as well as phenomena occurring during the process of friction, measured in most cases by the amount of wear and coefficient of friction.

Durability of products is decided by surface layers which, depending on the type of technological process applied, may have a thickness of orders of $1\ \mu\text{m}$ to several mm. The condition of the decisive surface layer is critical to wear resistance during the process of friction and to corrosion resistance. In the case of mechanical loading (especially fatigue resistance) as well as the action of corrosion (hydrogen corrosion), the critical role is played by the substrate, its condition and properties as well as the atomic relationship between the surface layer and the substrate.

For this very reason, while considering life expectancy of machine components and assemblies, one should take into account the system: substrate – surface layer. Attributes to such a system are: thickness of the surface layer – ratio of this thickness to the entire cross-section, ratio of surface hardness to core hardness and the state of residual stresses, usually compressive within the surface layer, relative to the state of stresses in the substrate, which are usually tensile. An incorrectly applied surface layer may cause formation of a structural flaw in the transition zone of the layer and may lead to crack initiation, especially by the fatigue mechanism (Nakonieczny, 1984).

The volume heat treatment and other processes belonging to surface treatment do not function independently, but are an important part of the general domain of surface engineering.

The surface engineering encompasses technology behind formation of surface layers, but also defines service properties of products, surface layer investigation methodology as well as design considerations for the substrate-layer system according to given service conditions.

Service properties of products, and hence the functionality of the surface treatment, may be assessed by defining the fatigue limit, wear resistance or corrosion resistance. Such evaluations are usually performed on specimens in laboratory conditions. However, most valuable information is to be gained from actual service trials. The costs of such investigations are, unfortunately, high and difficult to bear by smaller and medium size companies.

2. The concept of structural notch

Fatigue resistance of machine components is a function of their design, material and technological parameters as well as the type of loading in service conditions. When discussing the problem of fatigue resistance, one should discuss in detail the effect of these parameters, and in the case of loads, define fatigue characteristics, e.g. Smith's plots for different types of loading such as bending, tension and torque. These problems have been sufficiently dealt with in technical literature.

In the process of searching for methods of increasing fatigue resistance, there occur some constant elements, the implementation of which has a favorable effect on it. To these belong enhancing treatments such as thermal, thermo-chemical as well as surface work hardening.

In order to raise fatigue resistance, it is not sufficient to apply a chosen enhancement treatment, but rather of utmost importance is appropriate selection of the initial volume heat treatment prior to successive surface, thermal and work hardening treatment. The problem of enhancing fatigue resistance of machine components by technological methods does not consist of application of one chosen treatment, but of a cycle of successive treatments. The appropriate selection of these treatments affects the structural notch formed in the process of enhancement, which has decisive influence on fatigue resistance (Nakonieczny, 1984).

A structural notch occurs in all locations where, as a result of heat treatment (e.g. induction hardening), thermo-chemical treatments (carburizing, nitriding, etc.) or work hardening treatments (burnishing, shot-peening) of machine components, the layer formed in these processes has different physical-chemical properties than that of the core, due to a large gradient of property changes. The value of the structural flaw coefficient β depends on the type of material and parameters of technological processes which cause this structural notch to form. In other words, it depends on the heat treatment and surface hardening.

Industrially utilized thermal and surface work hardening treatments cause enhancement of fatigue resistance. Based on the research carried out at the Institute of Precision Mechanics (IMP), it can be accepted that as a result of implementation of such treatments, the fatigue limit (σ_{-1}) rises on average by 15 upto 30%. The enhancement of fatigue resistance is obtained by structural changes, strengthening and favorable distribution of residual stresses which are formed as a result of thermal and surface treatments.

Due to physical processes taking place within the material during the application of surface, thermal and work hardening treatments, changes in the microstructure and mechanical properties arise between the surface layer and the core of the material. The gradient of changes of physical-chemical properties depends on the selected technological process and its parameters. Numerous instances of fatigue cracks have been noted, whose origins were traced to the transition zone between the hardened surface layer and the substrate. Figure 1 shows, as an example, a fatigue fracture with the origin located under the hardened layer at the point where stresses are mounted. The mounting of stresses occurs as a result of residual stresses created during heat treatment and external stresses.

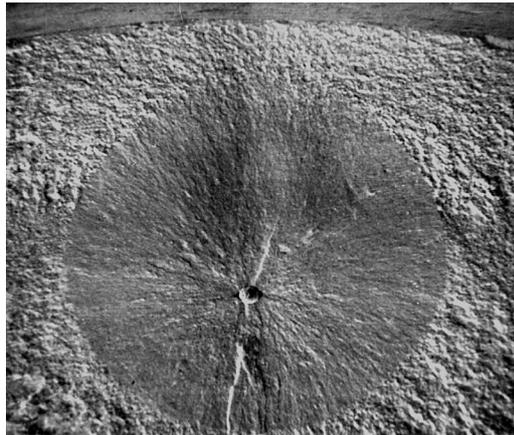


Fig. 1. Location of initiation of fatigue cracking on nitrided 40HM (4140) grade steel – $\times 100$

3. The role of substrate

For full evaluation of fatigue properties of thermally or mechanically treated materials, it is important to understand the mechanism of failure. Understanding this mechanism is possible by determination of the condition and mutual relationship between the substrate and the surface layer. Solution to this problem becomes possible through determination of the strength condition of the system: substrate–surface layer as a function of external loading.

The investigations were conducted on structural steels 40HM (4140) and 38HMJ (Nitralloy 135M). The steels were hardened and tempered prior to

nitriding at two temperatures: 550°C and 620°C (Nakonieczny, 1984; Babul *et al.*, 1996; Nakonieczny and Tacikowski, 1994).

Nitriding was carried out using two types of atmosphere, i.e. $\text{NH}_3\text{-NH}_3(\text{diss})$ and $\text{NH}_3\text{-N}_2$. In the nitriding processes, the atmosphere gas composition was varied, as were the time of nitriding (4 and 16 h) and nitriding potential – KN (from 1.65 to 4.8).

Fatigue resistance tests were carried out on the PUNN machine (manufactured by Schenck), applying rotational bending stresses with a notch ($\alpha = 1.02$). The specimen diameter was $\Phi = 5.88 \pm 0.02$ mm. The results of the fatigue resistance tests, metallurgical evaluation and process parameters are put together in Table 1.

Figure 2 shows microhardness traverses in the nitrided case for 40HM grade steel, while Fig. 3 shows the same for the 38HMJ grade.

The results of tests show that the tempering temperature has an effect on the properties of the nitrided case. The effect of the tempering temperature on the basic properties of the nitrided case depends on the steel grade. A higher increase of hardness (by about 50%) as well as of case depth is observed on the low alloy chromium steel 40HM.

Higher value of the fatigue strength limit can be observed for higher hardness of substrate.

The results of investigation shown in Table 1 that for 4140 steel and hardness 402 HV0.5 and 396 HV0.5 the fatigue strength limit have values 820 MPa and 840 MPa, respectively.

For Nitralloy 135M we cannot observe the same phenomenon. For this material, we have another parameters of the surface layer, especially hardness and depth at which residual stress changes the sign.

4. Fatigue characteristics after carbonitriding

In most modern methods of manufacturing it is recommended that the design stage consider different manufacturing technologies. In connection with that there is an urgent need to determine material characteristics, especially of materials after application of modern enhancing treatments. There also exists the need to develop calculation methods of fatigue resistance after thermal and thermo-chemical treatment. This, however, is the next stage of the activity, possible to carry out only when the basic fatigue properties of steel following a heat treatment will be known.

Table 1. Technological parameters and test results

Steel grade	Tempering temperature [°C]	Nitriding time [h]	Core hardness HV0.5	HV0.5 Hardness		Fatigue limit σ_{-1} [MPa]	Residual stresses	
				Max. on cross-section	On surface		At surface [MPa]	Depth at which stress changes sign [mm]
40HM (4140)	550	4	402	677	757	820	600	0.32
	550	16	396	642	757	840	650	0.52
	620	4	343	715	826	735	600	0.37
	690	16	343	343	642	745	900	0.55
38HMJ (Nitalloy 135M)	550	4	356	1030	1373	805	900	0.25
	550	16	343	1030	1227	785	600	0.48
	620	4	318	1030	1273	766	450	0.30
	620	16	296	1030	1304	810	800	0.45

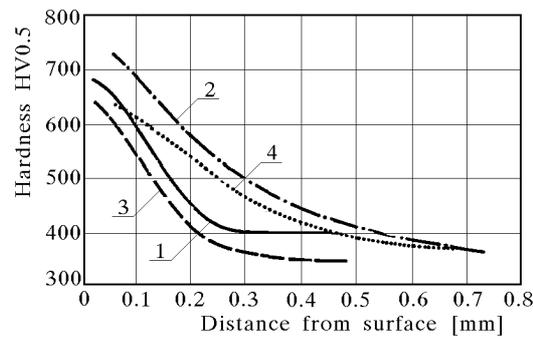


Fig. 2. Microhardness traverses across the nitrided case on 40HM (4140) grade steel; 1 – tempering temperature 550°C, time 4 h; 2 – tempering temperature 550°C, time 16 h; 3 – tempering temperature 620°C, time 4 h; 4 – tempering temperature 620°C, time 16 h

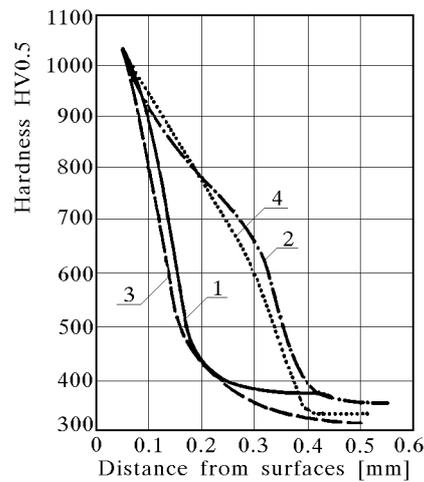


Fig. 3. Microhardness traverses across the nitrided case on 38HMJ (Nitalloy 135M) grade steel; 1 – tempering temperature 550°C, time 4 h; 2 – tempering temperature 550°C, time 16 h; 3 – tempering temperature 620°C, time 4 h; 4 – tempering temperature 620°C, time 16 h

The carbonitriding treatment is used for components exposed to lighter loads and subjected to wear as well as bending (Tacikowski and Nakonieczny, 1992; Nakonieczny, 1991a,b; Winderlich, 1990; Kogaev *et al.*, 1985). For those components, which are subjected during service to contact fatigue, the case depths are designed deeper. For the present series of tests, a case depth of 0.7 mm was selected.

The optimum microstructure of carbonitrided components is fine acicular martensite with a small amount of retained austenite and not containing coarse carbide precipitations (Taciowski and Nakonieczny, 1992; Nakonieczny, 1991a,b; Winderlich, 1990; Kogaev *et al.*, 1985).

Specimens prepared to meet the above conditions were subjected to rotational bending and one point bending fatigue tests. Such types of loading were selected based on the premise that bending is the most common method of loading during service as well as the fact that during bending one can observe the most favorable and strongest effect of surface strengthening.

Simplified Smith's curves were plotted to determine the fatigue resistance for at least three methods of bending (the Wöhler curve) as well as static strength and yield strength for a given type of loading and materials. The fatigue tests were carried out with the following coefficients of cycle asymmetry: $R = -1$; $R = 0.1$ and $R = 0.3$. The coefficients of 0.1 and 0.3 were selected to ensure possibility of running the tests only within the range of one sided bending stresses.

The fatigue characteristic was developed for a material in the quenched and tempered condition, as a reference, and for materials with a diffusion case, heat treated to the same condition as the only heat treated version. Once these values were known, the surface coefficient of strengthening was determined from the equation

$$m = \frac{\sigma_{-1}^{ww}}{\sigma_{-1}} \quad (4.1)$$

where

- σ_{-1}^{ww} – fatigue limit of the enhanced specimen
- σ_{-1} – fatigue limit of the reference specimen.

Bending tests were carried out on the Amsler machine. In order to obtain the bending effect on this machine, a prototype additional element was designed which, through a lever applies loading of the tested section of the specimen under a constant bending moment (Fig. 4). The frequency was 150 Hz. The tests were carried out to $N_G = 10^7$ cycles.

The material used in these tests was the 18HGT grade, normalized, with a fine grained ferrite-pearlitic microstructure.

Carbonitriding of specimens made of 18HGT grade steel was carried out at temperature 860°C in an endothermic atmosphere, enriched with ammonia and natural gas.

Metallurgical evaluations were carried out on 18HGT steel in the quenched and tempered only and carbonitrided conditions.

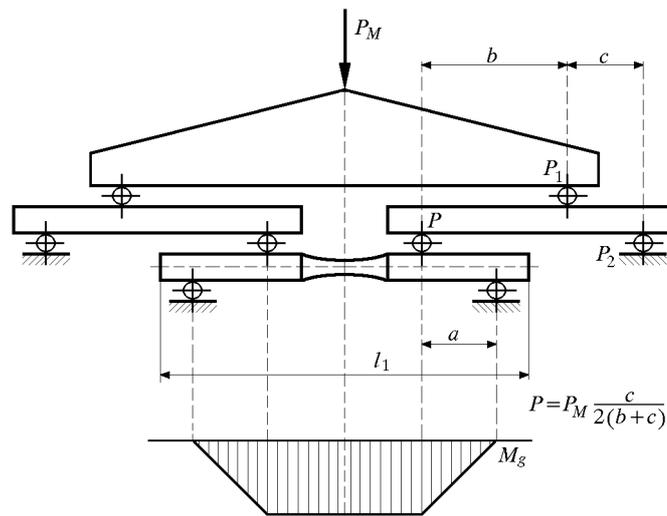


Fig. 4. Schematic of equipment for fatigue testing

In the normalized only condition, the specimens showed ferrite-pearlitic microstructure with very fine-grained pearlite (Taciowski and Nakonieczny, 1992; Olszański, 1977; Olszański *et al.*, 1979; Wyszowski, 1974).

The microstructure of specimens with diffusion cases was determined based on photomicrographs and microhardness measurements.

Specimens made of 18HGT steel, after carbonitriding and quenching with tempering exhibit in the subsurface zone a microstructure of tempered martensite (Fig. 5).

In order to determine the bending resistance, a static bending test was carried out. For the heat treated (normalized) only steel, it was not possible to obtain a static bending strength value because of ductility of the material. Only the yield strength was determined, and for the specimens it amounted to 822.8 MPa.

The static bending test carried out on carbonitrided specimens is shown in Fig. 6. In this case it was not possible to determine the yield strength, and only the elastic limit in the point A was established as 2289.8 MPa.

An analysis of results of the static tests delivers new data. The tensile plot for the carbonitrided material is characteristic for brittle materials. There is no necking and no elongation of the specimen. Similar behaviour was noted when the bending strength test was performed. In no case it was possible to determine the yield strength.

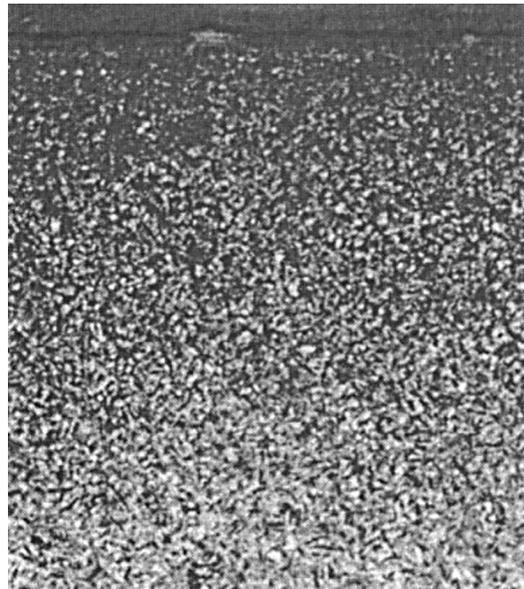


Fig. 5. Microstructure of the carbonitrided case on a specimen made of carbonitrided 18HGT grade steel. Etched by Nital – $\times 500$

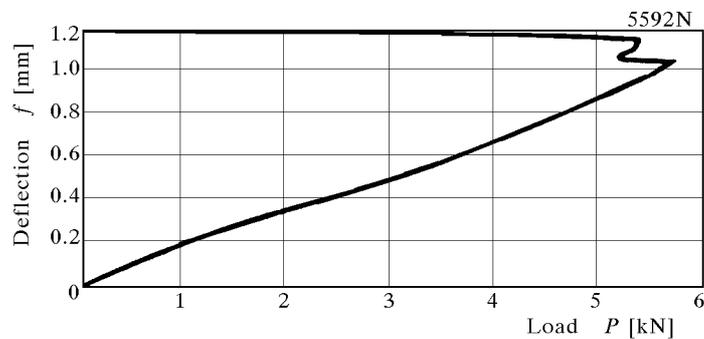


Fig. 6. Plot of the static bending test of a specimen of carbonitrided 18HGT grade

Based on fatigue tests for rotational bending, which were performed on specimens made of carbonitrided and heat treated (normalized) only material, it was possible to determine the coefficient of surface strengthening, i.e. the ratio of $m = \sigma_{-1}$ (with diffusion case) to σ_{-1} (with no case). For 18HGT steel after carbonitriding this coefficient was 2.48.

With the aid of results obtained in static and fatigue tests for the case of two and one side bending, simplified Smith's curves were plotted for the heat treated only material and for the carbonitrided material (Fig. 7). To plot the

chart, values of unlimited fatigue resistance were used from the Wöhler curves. The upper limit of the chart for the heat treated only material is the yield strength obtained from the bending test.

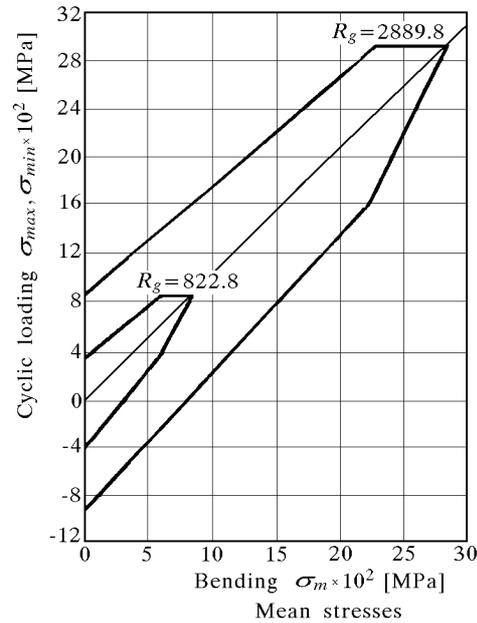


Fig. 7. Simplified Smith plot for 18HGT grade steel after normalizing (1) and carbonitriding (2)

The Smith curve for materials after thermo-chemical treatment differs from that for the reference heat treated only material because its upper limit is determined by the bending yield strength R_g (Fig. 7). A designer of the component, basing his design on the presently available tables containing data of the ultimate properties of the steel after hardening and fatigue properties for alternating stresses, creates a design, which consumes big amounts of the material. As a result of using values of σ_{-1} taken from catalogues, the strength of the assembly is compromised. The values of the fatigue resistance σ_{gR} (where $-1 < R < 1$) are much higher, which can be seen from the Smith plot.

5. Graphical pattern of fatigue fracture

Among the most important parameters describing the condition of the surface layer are, microstructure, degree of strengthening, state of stresses and

roughness. The said parameters depend on other important parameters such as texture, surface energy and chemical composition (Nakonieczny and Tacikowski, 1994; Nakonieczny, 1991a,b).

In engineering practice, usable properties such as tensile strength R_m and, fatigue limit σ_{-1} , may be determined as functions of mechanical parameters, i.e. hardness H , tensile strength R_m or surface roughness (Nakonieczny, 1991a,b; Olszański, 1977). Such correlations maintain their validity for a material homogenous throughout its cross-section (Fig. 8).

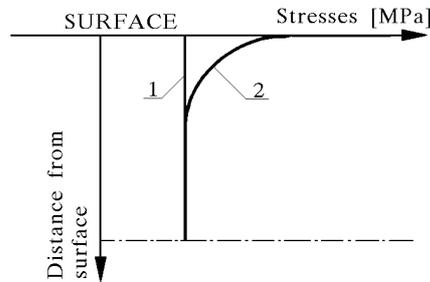


Fig. 8. Fatigue characteristics for a homogenous material (1) and non-homogenous (2)

For heterogeneous materials, e.g. ones that have been surface treated, such correlations cannot be applied directly.

The character of distribution of basic mechanical properties, i.e. hardness and residual stresses as well as their significant effect on fatigue properties of a material suggest that the distribution of the fatigue limit across the cross-section should be basically the same as that shown in Fig. 8.

The fatigue characteristic, which is shown by the distribution of the fatigue limit σ , is a function of the hardness H and residual stresses σ_r

$$\sigma = f(H, \sigma_r) \quad (5.1)$$

The method of designing a usable characteristic of the fatigue limit distribution was described in publications (Nakonieczny, 1991a,b; Kogaev *et al.*, 1985). In this paper, the influence of residual stress is not considered.

The distribution of stresses from extraneous loading constitutes a significant characteristic because it enables determination of the strength condition for the surface layer. Loading characteristics are typical distributions of stresses across the section of a component or specimen for the investigated types of loading, tensile-compressive, bending or torque. For smooth specimens, their determination does not present any problem. Some difficulties may arise when determining distribution of stresses in a notched specimen.

The distribution of stresses from extraneous loading for notched specimens in conditions of bending can be obtained from the following formula (Nako-nieczny, 1991b)

$$\sigma_{max}(x) = \sigma_n \alpha \left[1 - \left(\frac{2x}{d} \right) \right]^{3\alpha-2} \quad (5.2)$$

where

- $\sigma_{max}(x)$ – local stress at a distance from the surface
- σ_n – nominal stress
- α – coefficient of stress concentration
- d – cross-section dimension.

The knowledge of the fatigue characteristic as well as the loading characteristic allows one to determine the fatigue strength condition for any location on the component cross-section

$$\sigma_{-1} \geq \sigma_i^{oz} \quad (5.3)$$

where

- σ_{-1} – fatigue limit at any location on the specimen cross-section
- σ_i^{oz} – value of stresses from extraneous loading at the given location i .

The effect of heat treatment of the core on the fatigue resistance is shown in Fig. 9. It is seen that the rising of the core hardness moves the fatigue resistance characteristic, i.e. the distribution of the strength limit value across the section in the direction of higher stresses.

Data to determine the characteristics shown in Fig. 9 are put together in Table 2.

Table 2. Values of the fatigue limit across the specimen section

Fatigue limit σ_{-1} [MPa]	Experimental results (Table 1)		Theoretical results (Eq. (5.4))	
	Tempering temperature			
	550°C	620°C	550°C	620°C
Core	613	550	618	550
At surface	–	–	852	819
At location of fracture initiation	820	735	618	550

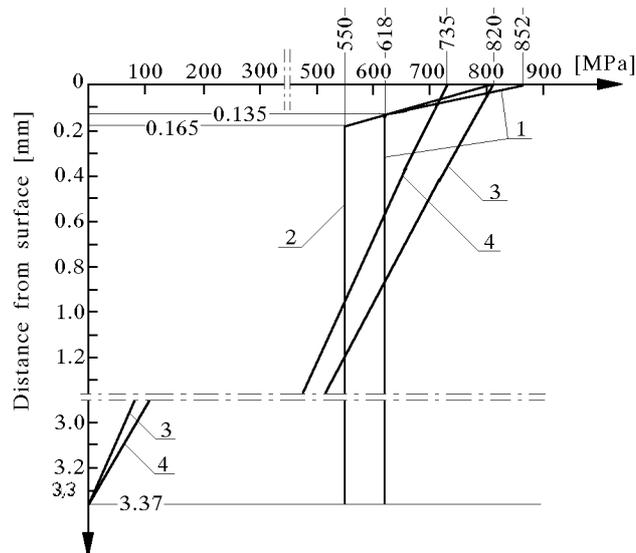


Fig. 9. Distributions of the fatigue limit – curves 1, 2; residual stresses – curve 3 and 4, 40HM (4140) grade steel (theoretical analysis)

To calculate the fatigue limit, the following formula was used

$$\sigma_{-1} = 1.98HV - 0.0011(HV_i)^2 \quad (5.4)$$

where HV is the Vickers surface hardness, HV_i – Vickers hardness at the i th location on the cross-section.

The relationship is valid for the hardness range $340 \leq HV \leq 900$ (Winderlich, 1990).

A significant increase of the fatigue limit (to 820 MPa) with a tempering temperature of 550°C and up to 735 MPa with a tempering temperature of 620°C , relative to prior values of 618 MPa and 550 MPa, determined at the location of fatigue crack initiation (Fig. 1) (0.5 mm from the surface on average) should be interpreted as a favorable effect of compressive stresses in the nitrided case.

It follows from Fig. 9 that the initiation of fatigue cracking of nitrided cases (compare with Fig. 1) takes place under the surface because stresses from extraneous loading locally exceed the value of the fatigue limit and, in accordance with line 3 in Fig. 9, material decohesion must occur.

6. Summary

Modern structural materials do not have to be homogenous throughout their section. A great number of steels, plastics and other metallic materials call for surface enhancement, due to constant quest for decreasing material and energy consumption as well as improving properties. Surface layers are, in the majority of cases, superficial hardened layers, formed by thermal and thermo-chemical treatment or other enhancement technologies such as surface work hardening and anti-corrosion coatings.

Tests on material properties after heat treatment show that the achievement of desired service properties is connected with appropriate selection of parameters not only of the final thermo-chemical treatment but also of the prior volume heat treatment. In the case of nitriding, this technology is usually preceded by quenching and tempering at a temperature minimum 20°C above the subsequent nitriding temperature. The initial hardening by quenching and tempering is critical to the core hardness and to properties of the nitrided case, and affects the fatigue resistance of the material after nitriding.

Analysis of fractured surfaces of nitrided specimens exposed to service in conditions of rotational bending revealed that the weakest location on the specimen cross-section is the zone of transition of the nitrided case to the core. The method of designing surface cases enables the explanation of the root cause of initiation of fatigue cracks under the nitrided case. The fatigue limit in the cross-section of the specimen is described as a function of microhardness and residual stresses. The initiation of fatigue cracks takes place in the location where stresses from extraneous sources exceed the value of the fatigue limit, which is obviously in agreement with conditions of strength. The favorable effect of core hardness on fatigue resistance was observed.

It was established that fatigue resistance is significantly affected not only by compressive stresses but also by tensile stresses.

Among parameters describing the state of residual stresses, the distribution of stresses and the value of the residual stresses are of more significance.

Models of surface layers described in literature are difficult to implement in industrial practice. There appears a necessity for creation of such a model of the surface layer. It could be described by parameters which can be utilized in strength calculations and which would allow its application in instances of different types of extraneous loading, depending on the type of service of the component. This model could become the basis for predicting the state of the surface layer, based on required usable properties of machine components. The work on such a model is carried out in two directions:

- based on experimental description of the state of the surface layer through hardness traverses, residual stress distributions and distributions of element concentrations, e.g. carbon and nitrogen,
- based on description of the material through theory of elasticity and solution to constitutive equations by numerical methods.

The design of the surface layer criterion of failure by fatigue is based on the comparison of the local fatigue resistance with local stresses occurring at critical locations in the investigated component.

Contemporary machines and designs should be characterized by required life and reliability, featuring sufficient life between overhauls, depending on operating conditions, while at the same time fulfill requirements of ecology and ergonomics.

Such parameters should be attained concurrently with reduction of a material and energy consumption during manufacture and service.

This task may be achieved only when at each stage of the product life, i.e. study phase, design, manufacture, service and recycling, modern computational methods will be implemented along with modern technology and proper service conditions.

The implemented computational methods enable products to be designed according to strength criteria, somewhat less often – tribological and least often – pertaining to corrosion.

Contemporary machine components and assemblies are subjected during service to joint strength interactions, tribological and corrosion hazards. On the other hand, the implemented computational methods enable products to be designed for one selected mode of failure.

In machine design there are many parts (crankshafts, threaded joints, springs) which during service are concurrently exposed to different types of failure hazards: mechanical, tribological or corrosive. Similar elements of structures (bridges, masts, cables, earth – moving and mining machines) are exposed to concurrent hazards of fatigue – type stresses and corrosion (Nakonieczny, 1991a,b).

Classical strength or tribological calculations do not take the factor of time into account. During service, due to processes of fatigue, tribological or corrosive deterioration, there occurs a change in properties of a system being calculated. Tribological and corrosive processes cause a change in the geometry and surface condition of a component. This, in turn, causes a change in the state of stresses in working systems, affecting their life and reliability.

It stems from the above that the development of failure criteria taking into account joint effects related to damage accumulation due to working of

alternating loads, wear by friction and corrosion, is a very important task because the determination of failure criteria will enable proper selection of surface layers for given service conditions.

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Pękanie zmęczeniowe warstw azotowanych i węglazotowanych

Streszczenie

W artykule opisano zjawisko pęknięcia zmęczeniowego warstw azotowanych i węglazotowanych. Właściwości zmęczeniowe opisano przez analizę właściwości podłoża i warstwy wierzchniej. Określono wpływ mikrotwardości podłoża na właściwości zmęczeniowe warstwy azotowanej. Prezentowane są wykresy Smith'a dla warstwy węglazotowanej. Przedstawiono graficzną metodę dla przewidywania miejsca inicjacji pęknięcia zmęczeniowego. Przedstawiono kierunki badań właściwości tribologicznych i korozyjnych.

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