STRUCTURAL ASPECTS OF FRACTURE IN NITRIDED LAYERS

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ABSTRACT

Effects of nitrided layer depth and especially of compound layer thickness on fatigue life and strength has been experimentally studied. Fractographic analysis revealed the close relationship between mode of fracture initiation and fatigue life and strength. More thick compound layer decreases fatigue resistivity. Similar, more significant negative effects were obtained by application of annealing after nitriding procedure in spite of relatively positive effect on structure of nitrided layer (increased depth of nitriding, removed compound layer).

KEYWORDS

Fatigue, plasma nitriding, annealing, compound layer, diffusion layer crack nucleation, inclusion, crack propagation.

INTRODUCTION

Modern technologies of micro pulse plasma nitriding enable relatively exact controlling of nitriding procedure. Layers of different depth of nitrogen penetration and of different phase compositions can be realised. An important parameter, determining namely fatigue strength of nitrided parts, is thickness of surface compound layer, usually composed of γ' and ε nitrides. In this relation, effect of annealing after nitriding process has been widely studied [1]; its application increases depth of nitrogen penetration and simultaneously decreases thickness of compound layer. The presented paper describes some selected results of experimental research works [2] that were realised with two goals:

a) To study effect of compound layer thickness
b) To determine influence of annealing on the fatigue properties of studied steel.

MATERIAL AND NITRIDING TECHNOLOGY

Steel CSN 15340 (37CrAlMo6) was used as the experimental material. Chemical composition of steel is in the table 1. The introductory heat treatment of steel was the following:

- normalising:	900 °C / 25' / air cooling
- quenching	930 °C / 25' / oil
- tempering:	650 °C / 40'/ oil

Table 1. Chemical composition of steel CSN 15 340

Element	C	Mn	Cr	Mo	V	Cu	Al	W	Si	P	S
weight %	0.371	0.324	1.46	0.14	0.01	0.048	0.88	0.109	0.248	0.007	0.009

Parameters of micro pulse plasma nitriding process are presented in the Table 2. Three technologies, denoted (a), (b) and (c) were used to obtain 20 specimens for each technology; another 28 specimens for fatigue and tensile tests were tempered only.

Table 2. Plasma nitriding procedure

	Required properties	*STEP*	t_{in}. [°C]	*Time* [h]	*Atmosphere* H_2	N_2	CH_4	*Press.* [mbar]	*U* [V]	*Puls* [μs]
(a)	***h*nitr : 0.2 mm** *comp. layer:* +	cleaning	510	0:30	20	2		0.7	800	100
		nitriding	515	8:00	24	10	0.4	2.6	530	120
		annealing	-							
(b)	***h*nitr : 0.4 mm** *comp. layer:* +	cleaning	530	0:30	20	2		0.7	800	120
		nitriding	535	26:00	24	10	0.4	2.6	530	150
		annealing	-							
(c)	***h*nitr : 0.4 mm** *comp. Layer: -*	cleaning	530	0:30	20	2		0.7	800	100
		nitriding	535	24:00	30	6		2.6	535	150
		annealing	535	4:00	30	-		2.6	550	150

Note: $h_{nitr.}$ – nitrided layer depth (total); t_{in} – temperature of the steel charge

PRINCIPAL PROPERTIES AND STRUCTURE OF NITRIDED AND TEMPERED SPECIMENS

Survey of basic properties of nitrided specimens (maximum and minimum values for each technology) is in the table 3. Strength properties of tempered steel before nitriding are shown in table 4.

Table 3. Properties of nitrided specimens

nitriding technology	$R_p0.2$ [MPa]	(R_{eH}) [MPa]	R_m [MPa]	A_t [%]	HV_{max} (surface)	HV_j (core)	h_{nitr} [μm]	h_l [μm]
(a)	885	-	1040	5.44	1205	299	180 ÷ 240	1.26 ÷ 2.98
	858	-	1020	4.89	1205	296	190 ÷ 200	0.99 ÷ 2.75
(b)	871	(879)	1020	5.23	1101	291	200	1.80 ÷3.96
	850	(858)	996	5.28	1112	305	180 ÷ 200	1.89 ÷ 3.92
(c)	785	(774)	993	3.56	1112	287	390 ÷ 400	-
	758	(758	981	3.22	1112	281	390 ÷ 420	-

Note.: h_l - thickness of the compound (white) layer, h_{nitr} - nitrided layer depth (total)

Tab. 4. Strength after tempering

Technology of nitriding	R_{eH} [MPa]	R_m [MPa]
(-)	839	954
(a)	861	974
(b)	819	944
(c)	840	953

In comparison with strength values of steel before tempering, values of yield point and ultimate tensile limit R_m are moderate higher for technologies (a) and especially (b), but both values were significantly decreased for nitriding technology (c). Ductility of nitrided specimens was low for all technologies, only about 5 %.

Irregularities in stress-strain curve (Fig.1) of nitrided specimens, evaluated by software of testing machine as the sharp yield point R_{eH} are more probable results of nitrided layer fracturing. This event takes place approximately at stresses corresponding to the proof stress $R_p0.2$ of the base material (core). Deformation approximately 0.2 % seems to be equal to the ductility of nitrided layer.

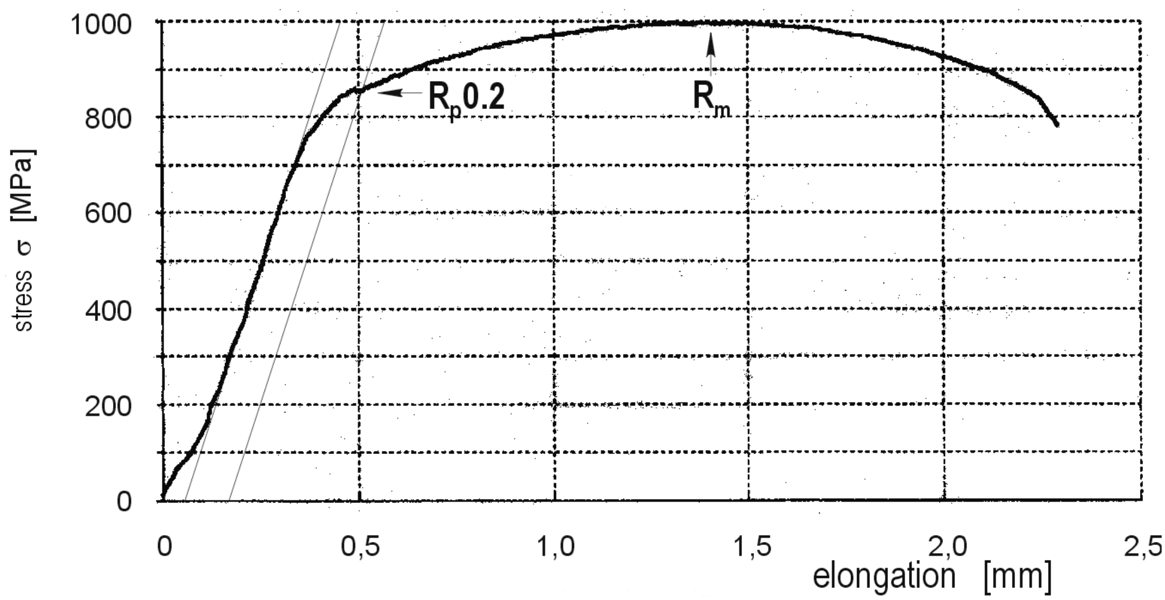

Fig. 1. Stress - strain curve for technology of nitriding (a)

Depth of nitrided layer was determined from the hardness profile measured by LECO microhardness tester. Typical profiles for each technology are on Fig. 2.

Thickness of compound layer (see table 3) was measured on metalographs, that are presented in figures 3a), 3b) and 3c).

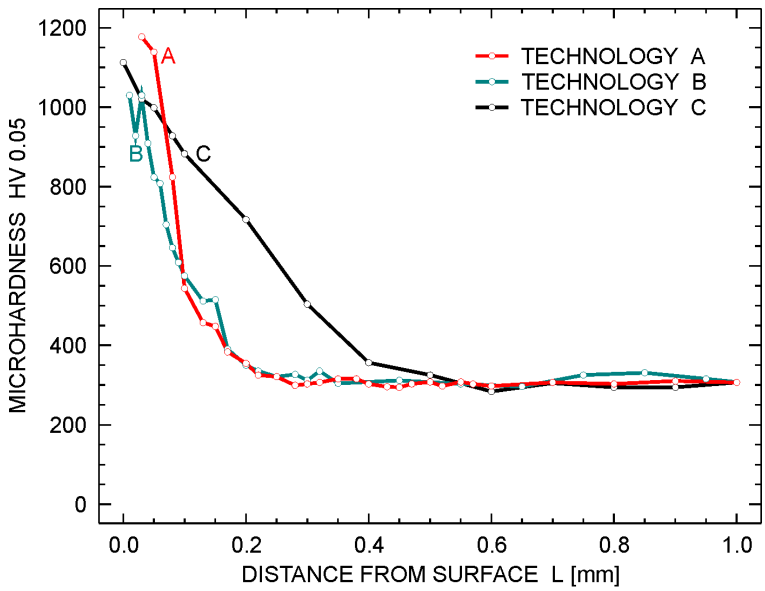

Fig. 2. Microhardness profiles of technologies (a), (b) and (c)

Fig. 3a Technology (a) 500x

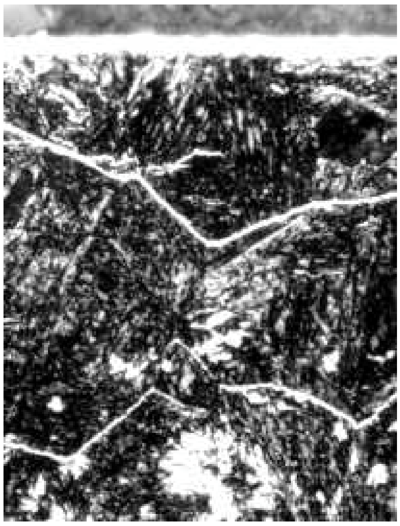

Fig. 3b Technology (b) 500x

Fig. 3c: Technology (c) 500x

Results of individual methods of nitriding on microstructure and principal mechanical properties of layer can be summarised as follows:

- **technology (a)** enabled to obtain the required properties, it means total depth of nitriding 0.2 mm and low thickness of compound layer of γ′ and ε nitrides (from 1 up to 3 μm). Surface hardness is the highest in comparison with other used technologies, it achieves 1200 *HV* 0.05.

- **technology (b)** did not produce the expected results. More than 3 times longer time of nitriding with respect to technology (a) did not increase depth of nitrided layer, but caused increment in the thickness of compound layer (about twice - from 2 up to 4 μm). Presence of relatively coarse compound layer is visible in the hardness profile, too. Results of chemical composition and phase analysis are a little contradictional. Profile of chemical composition of surface, measured by means glow-discharge spectral analyser LECO GDOES SA-2000 shows on increased N in the surface (aprox. 11.0 % N). Significantly, the surface is enriched with carbon (5 % C to depth 1.5 μm under surface). Amount of Cr is also higher (about 2 %). But, rentgenographic phase analysis declared presence of carbide Fe_3C only and small fraction of nitride γ' $(Fe_3Ni)N$. Moreover, these results are not

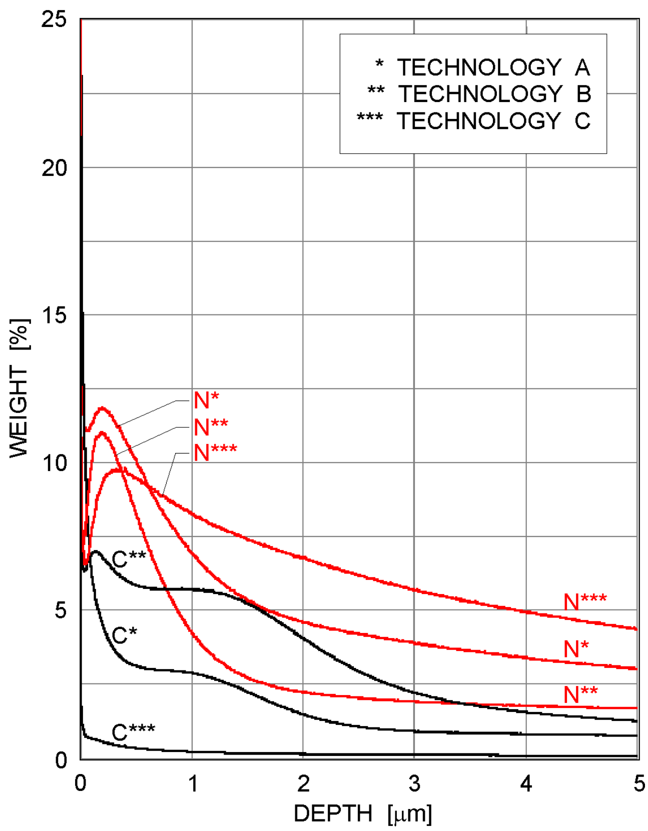

Fig. 4. Profiles of carbon and nitrogen for technologies (a), (b) and (c)

supported by microhardness tests results, measured value 1050 *HV* 0,05 is lower than for technology (a).

- **technology (c):** requirement to decrease compound layer thickness was solved by modification of nitriding atmosphere (lower amount of N_2, no CH_4) and by application of subsequent annealing. This procedure really minimised the thickness of compound layer, but induced an extensive process of N diffusion. Depth of nitriding increased up to 0.45 mm. Profile of N inside the part of layer is in Fig. 4. Sudden drop of Ni content was also detected; the increased amount of Cr was kept in surface as the rest of compound layer. Maximum hardness is on surface and then hardness continuously lowers, any „plateau of high hardness" in the area of compound layer are not visible. It could be supposed, that these effects have to be joined with changes in phase composition and with the change in the level of internal stresses.

RESULTS OF FATIGUE TESTS

Fatigue tests of steel 15 340 in tempered and nitrided states were performed on resonant testing machine Rumul – Mikrotron 25 kN. Load asymmetry ratio was 0.1 (tension – tension loading), frequency of loading approx. 80 Hz.

Results of tests are presented in Fig. 5 in form of S-N curves, plotted for maximum stress in loading cycle. Dashed lines, denoted (-) show the level of fatigue limit and endurance of steel 15 340 in tempered state. Lines, marked by symbols (a), (b) and (c) correspond to different nitriding technology of the same notation as in the table 2. Moreover, levels of proof stress (R_p0.2) are marked out in diagrams.

Discussion of results:

- Fatigue limits of specimens, nitrided by technology (a) or (b) are higher than fatigue limit of only tempered specimens. Thickness of compound layer has the significant effect, nearly two times thicker compound layer obtained by technology (b) decreases values of maximum stress for fatigue limit with 45 MPa (approximately 5 %).
- Application of annealing (technology c) strongly reduces fatigue limit and fatigue endurance despite of increased depth of nitrided (diffusion) layer. Possible reason of this behaviour is reduction of internal stresses level. Experiments, done in our department showed, that the presupposition of compressive stresses in nitrided specimens is not generally truthful. Type of internal stress (compressive or tension) depends on chemical composition of nitrided steel and on the nitriding procedure [1]. This diversity of internal stress levels was also found for annealed specimens, but with dominating tension stress type. This fact may be also reason of lower proof stress of specimens (c).
- Anomalous behaviour, typical for super long – life regime [3] can be joined with technologies (a) and (b). Fatigue life line of S-N diagram separates into two parts – region of very low fatigue life (to 10^4 cycles) and region of long fatigue lives (over 10^5 cycles). Each of mentioned two regions corresponds to characteristic mode of fracture initiation, as will be discussed in the next chapter.
- There is variance in fatigue process of specimens with different thickness of compound layer (technologies a, b), what is evident from variant slope of S-N curve in short – life regimes.

- One reason of above mentioned anomalies could be fact, that the level of loading corresponds to the level of nitrided layer strength. It causes fracturing of compound, fully nitridic layer and initiate the consequent fatigue fracture.

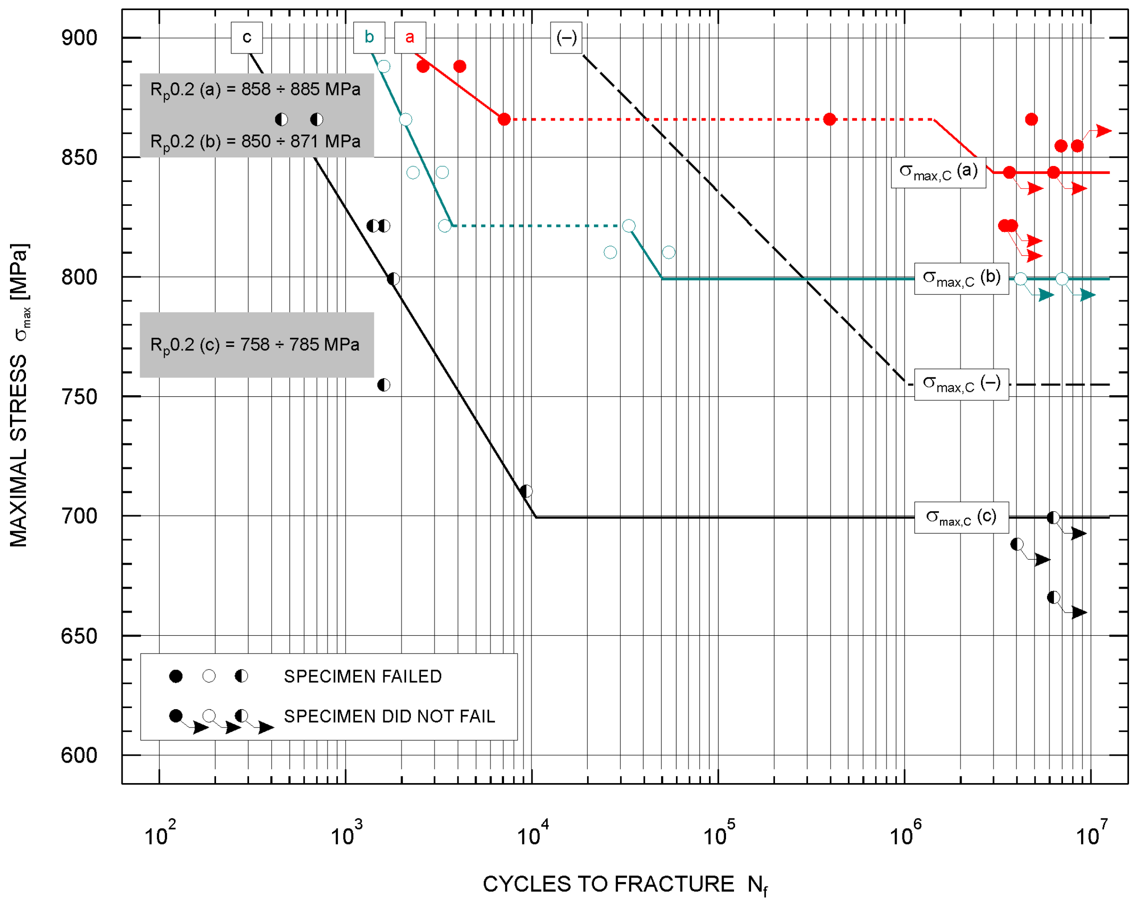

Fig. 5. S – N curve of steel 15 340; (-) tempered state, (a), (b), (c) – technologies of nitriding (Table 2)

FRACTOGRAPHIC ANALYSIS

Fractography was used as a tool for explanation of rather different fatigue lives at same stress levels, what was typical for technologies (a) and (b).

Nitriding technology (a)

Specimens, that were loaded to maximum stress 865 MPa were analysed. Fatigue fracture of specimen, which was broken after 4.8×10^6 cycles, initiated under nitrided layer on inclusion, consequent primary propagation of crack formed typical "fish eye" – Fig. 6a. This manner of nucleation on inclusion was described in [4], [5]. It is interesting, that in analysed case the initiation process is located below the layer, which is extensively cracked – Fig. 6b.

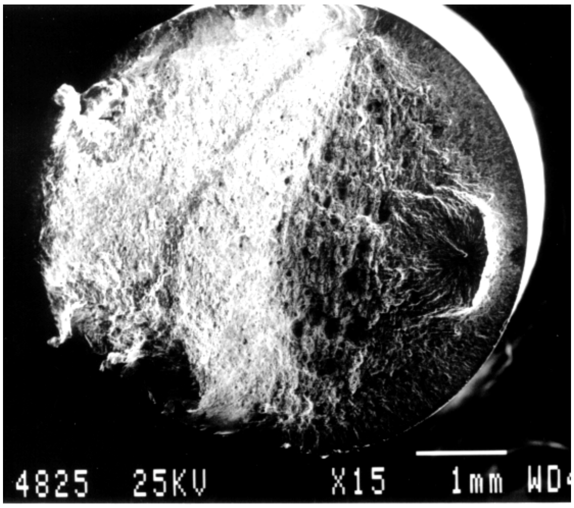

Fig. 6a. Fracture surface - technology (a) ($N_f = 4.8x10^6$ cycles)

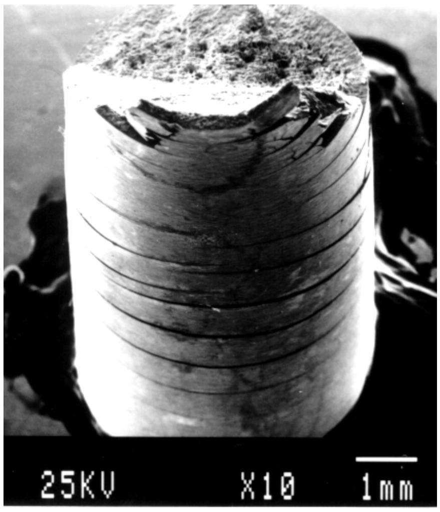

Fig. 6b. Crack in nitrided surface of the same specimen as in Fig.5a

Fatigue fracture process in specimens, that were broken after small number of cycles (up to 10 000), started on the surface from cracked nitrided layer (Fig. 7a, b).

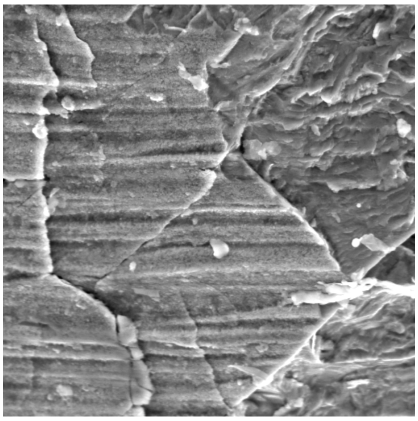

Fig. 7a. Cracking in the surface – techn. (b)

Fig. 7b. Fracture surface ($N_f = 7800$ cycles)

Technology (b)

The similar discontinuity can be observed in S-N curve of specimens, nitrided by technology (b), differences between numbers of cycles to failure are smaller then for technology (a). Fractographic analysis of specimens of different fatigue life (3 400 and 33 100 cycles), loaded to maximum stress 821 MPa does not ascertain any variances in fatigue fracture mechanism. In both cases, the fracture nucleated from cracked surface nitrided layer. Fracture morphology in this area has strong relation to microstructure of layer (Fig. 8); presence of intergranular net of nitrides is evident. In the depth about 100 to 250 μm, the

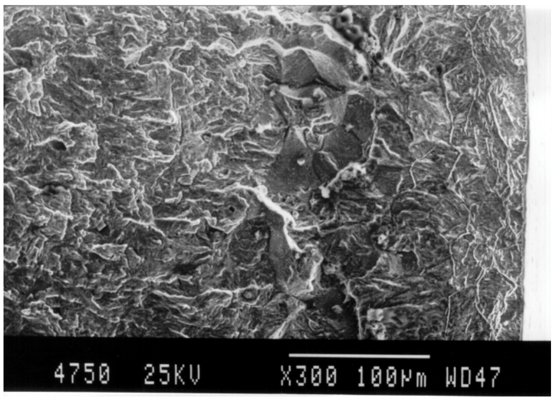

Fig. 8. Fracture morphology at surface of specimen – technology (b)

field of intergranular facets is visible. In some degree of simplification, the distance of this layer from surface is in inverse proportion to fatigue life of specimen. In any case, the fatigue life of specimen can be correlated to the period of long fatigue crack propagation; short initial crack prolong the stage.

CONCLUSIONS

The following complex conclusions can be deducted from experimental result:

a) The principal factor, influencing fatigue strength after nitriding is character of internal stresses. Decrease of primary compressive stresses (or transformation on tension stresses) by annealing treatment considerably lowers the fatigue limit of nitrided specimens.

b) Compound layer is an adverse factor; its increasing thickness decreases both fatigue limit and fatigue life.

c) Mechanism of fracture initiation is the decisive factor; in the case of nucleation from surface cracks in nitrided layer the fatigue life is comparable with the period of long fatigue crack propagation. This way of fracturing is often for loadings with tension mean stress.

d) Long fatigue life is consequence of fracture initiation under nitrided layer, usually on inclusion. For maximum cycling stresses, corresponding to the proof stress levels, but both discussed kinds of nucleation can occur.

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