SHEAR LIP FORMATION IN NITRIDED BLUNT NOTCH THREE-POINT DIE STEEL SPECIMENS

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ABSTRACT
Steel components are surface heat treated to enhance their wear and fatigue resistances. Nitriding and nitrocarburizing are among the most adopted surface treatments. Yet, these treatments cause the reduction of impact resistance and low cycle fatigue properties. A complete understanding of the reasons leading to the brittleness of nitrided and nitrocarburized components still lacking, results of researches leading to a detailed fracture mechanism sequence are presented.

1 INTRODUCTION

It is known that surface treatments such as nitriding or nitrocarburizing cause an increase of both wear and fatigue resistance in steel mechanical components. Moreover, they are able to reduce friction while increasing surface hardness. By nitrogen additions to the surfaces these treatments cause the formation on the surface of nitrided layers in which γ' and ε phases of the metastable Fe-C-N phase diagram (Fig. 1) are present, their relative amount being controlled by the nitrogen atoms quantity and therefore by the nitrogen potential of the selected process.

1.1 Mechanical properties of surface nitrides

Characteristics of the above phases in respect to wear and friction are markedly different; γ' is hard, but does not reduce the friction coefficient in a significant manner, whereas ε is able to substantially reduce the friction coefficient, but has an hardness which varies dramatically with the carbon content, passing from low values (220 HV ca.) at no carbon content to almost 800 HV in the vicinity of 3 wt. pct. carbon (1) (Fig. 2), irrespective of the N content.

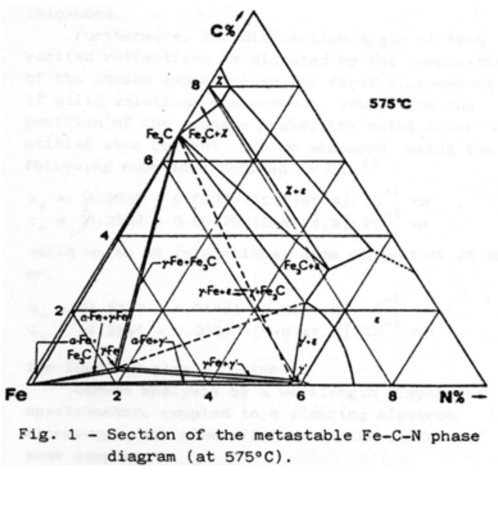

Fig. 1 – Section of the metastable Fe-C-N phase diagram (at 575°C).

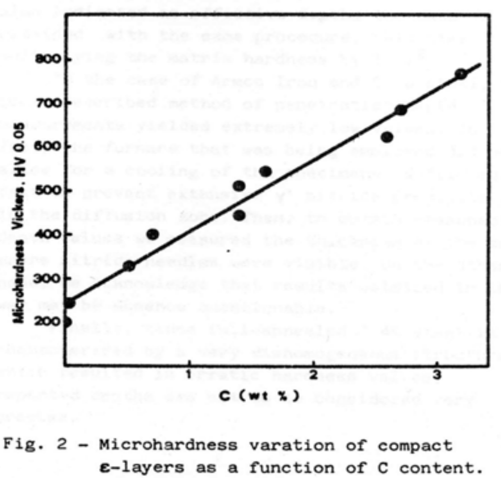

Fig. 2 – Microhardness varation of compact ε-layers as a function of C content.

Conversely, the ε-phase, which is very brittle when it contains only nitrogen atoms in interstitial solid solution, exhibits an improved toughness as the carbon content increases. Such a behavior, which contradicts usual metal alloys mechanical properties pattern, has not yet been explained.
Even considering that upon nitriding a steel part some carbon diffuses from the interior to the surface layer, thus causing some carbon intake by the surface ε nitrides, the above considerations explain why nitrocarburizing processes are constantly replacing nitriding treatments. It is also said that the formation of some Fe_3O_4 in the surface layers improves wear resistance; it can be obtained either by post-nitriding quenching treatments or by using oxidants directly during the surface treatment.

1.2 Mechanical properties of nitrided and nitrocarburized workpieces

The surface layer is followed by a subsurface layer, characterized by the diffusion of N atoms towards the interior of workpieces. Subsurface layer characteristics may enhance either wear or fatigue resistance. Nitrogen atoms diffusion causes the formation of matrix strengthening precipitates, whose frequency increases with the content of nitride stabilizing elements such as Al, Cr, Mo, V, Mn, and Si in the steel, thus strongly affecting subsurface hardness; the higher the subsurface hardness the more the mechanical component can withstand surface pressure during wear.
As it concerns fatigue resistance, different considerations pertain to the cases of high or low cycle fatigue.

1.2.1 High cycle fatigue
The intrusion of carbon and nitrogen atoms in the lattice of metal atoms causes the establishment of a residual compressive stress pattern in the directions parallel to the surface, highly beneficial from the point of view of fatigue life; the thicker the interstitial atoms diffusion layer, the higher the improvement of the endurance limit, σ_d.
Lessell's law (2), as revisited by Bell and Loh (3), gives a mathematical rendering of the above reasoning for nitrided cylindrical bars and is hereto displayed with a slight modification which takes into account results by Cowling and Martin on shallow diffusion layers (4);

$$\sigma_{d,t} = \underline{D}/(\underline{D}-2\underline{d})\cdot h\cdot{}_{d}\sigma_{,nt} \qquad (1)$$

In the above formula $\sigma_{d,t}$ e $\sigma_{d,nt}$ represent the endurance limits under rotating bending for treated and not treated steel bars, respectively; D̲ and d̲ are the bar diameter and the nitrogen diffusion layer thickness, whereas h is a constant introduced by the author (5), whose value can be set at 1.25 to better interpret experimental results on samples with thin diffusion layers.
Processes that allow the attainment of thicker diffusion layers at equal treatment times are therefore preferable. Nitrogen diffusion path length is reduced by increased amounts of precipitate forming elements and is enhanced by higher nitrogen potentials of the process. A simplified formula to interpret the above considerations, due to Mortimer, Grieveson, and K.H. Jack (6) and later applied by D.H. Jack and Stoney (7), is given below, again indicating with d̲ the diffusion layer thickness and identifying with t the treatment duration;

$$\underline{d}^2 = \frac{2\cdot(N)\%at.}{r\cdot(X)\%at.}\cdot\mathcal{D}\cdot t \qquad (2)$$

$\mathcal{D}$ is the nitrogen diffusion coefficient, (N)%at. is the atomic percentage of nitrogen dissolved at the medium-metal interface and r is the ratio of atomic masses of nitrogen and of a precipitate forming element. Finally, (X)%at. is the at. pct. of the same element as originally present in solution in the steel. In the case of the presence of multiple precipitate forming elements, (X)% at. is their total at. pct. and r is to be averaged.
The higher the nitrogen potential of the nitriding medium the thicker the diffusion layer up to the time a continuous surface layer is completely formed. From that moment on the diffusion layer growth rate is usually reduced, since it is then controlled by the nitrogen concentration at the interface between the surface layer and the ferritic matrix (7). It has been seen that nitrocarburized surface layers in which the ε solid solution predominates, allow a higher growth rate of the diffusion layer in the above described second stage in respect to the situation attained in simple nitriding. Such a finding can be rationalized hypothesizing a higher N potential at the interface when ε replaces γ' at the contact with the ferritic matrix.
It has also been seen that the duration of the first stage, in which the diffusion layer growth rate is more vigorous, is shorter in nitrocarburizing processes than in nitriding ones. It is then advisable, while surface treating dies, to begin with a nitriding first stage, followed by a nitrocarburizing second one.
Nitrocarburizing of extrusion dies can be effected either by salt baths containing alkaline cyanates and cyanides, or by glow discharge with atmospheres containing methane in addition to nitrogen and hydrogen, or by gas atmospheres of appropriate compositions.
Only by the gas treatment it is possible to easily divide the process in two stages by changing nitrogen and carbon potential of the treatment medium during the process. Furthermore, the gas process can be easily monitored and guided by continuous nitrogen potential control. Industrial treatments rarely use such an option.

1.2.2 Low cycle fatigue

Failure after a limited amount of strikes as sometimes seen in forging dies has not been studied extensively, nor modeling of fracture nucleating steps has yet been reached. A comprehensive study of the problem has been undertaken in the author's department (8, 9), also to relate the fracture process to the microstructure.
Fig. 3 depicts the microstructure of a nitrocarburized steel die; the relative extents of the top carbonitride layer and of the subsurface diffusion layer are clearly visible. White elongated precipitates, mainly extending in directions parallel to the surface, may also easily be detected. Contrary to the usual belief, they are not nitrides, but carbides of the cementite type (10). They may act as crack nucleating sites. Cracks may extend by fatigue to the surface, thus causing premature die failure.

2 NITRIDED SAMPLES FRACTURE MECHANISMS AND MODEL

Charpy-V type specimens fabricated employing both a traditional die steel (AISI H-13) and a newly developed low alloy one (0.34%C, 3% Cr, 0.90% Mo, 0.21%V) have been impact tested and their fracture surfaces examined both in the as-hardened (8) and in the as-hardened and surface heat-treated state (9); both gas nitriding and salt nitrocarburizing were industrially effected and different results analyzed.
It is known that SEM fractography gives the best clues to the effect of the microstructure on toughness. Therefore, a systematic fractographic analysis was performed on Charpy-V notch broken specimen halves, since, up to now, a complete critical assessment of fracture surfaces resulting from nitrided and nitrocarburized samples has never been performed.

First experiments showed that brittle fracture initiation in the top layer is followed by shear fracture along arcs of slip lines with final mode-I rupture completing in the bulk the separation process (9).
To have a better insight, series of blunt U-notch, plasma nitrided hot-work die steel samples with increasing notch root radii have been subjected to impact test and their fracture surfaces examined by scanning electron microscopy to distinguish between fracture morphologies in the thin top layers, along slip line arcs and in the bulk. Fractographic results were taken into account to rationalize the entire fracture phenomenon (11). Fracture appearance needing to be assessed by special SEM observation, specimens were mounted in the vacuum chambre so that a view tilted in respect to the overall fracture surface could be obtained. In fact, observation from a direction normal to the fracture surface had given the idea of the formation of a double arc shear lip (Fig. 4).

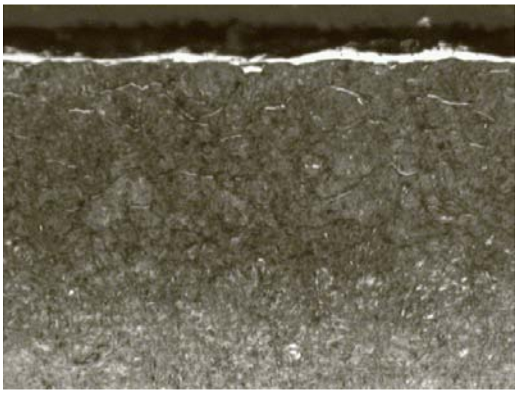

Fig. 3 - Microstructure of surface layers of a salt nitrocarburized AISI H-13 steel; nitrided edge on top.

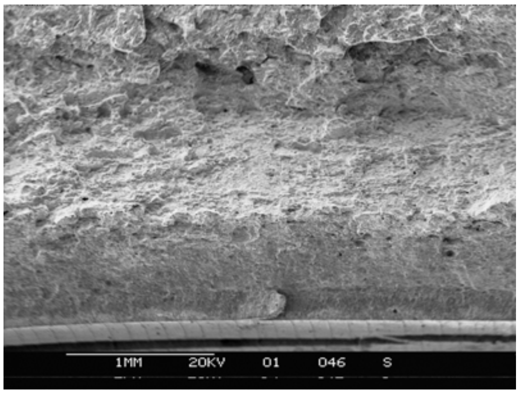

Fig. 4 - Normal view of a broken plasma nitrided low alloy steel sample (Charpy-V notch, ρ = 3.0 mm); nitrided edge on bottom.

Fig. 5 reports a tilted view of a broken AISI H-13 steel specimen half. It is here evident that fracture develops in a direction forming an angle with the notch centerline and proceeds as a flat fracture in radial direction from the notch center up to a depth of about 50 μm. Then an arc shaped shear lip develops up to a depth of about 200 μm.
Indeed, two shear fractures develop almost concurrently at equal angles from the notch centerline, starting from radial cracks equally positioned in respect to the notch geometry. One of the two fractures reaches first the region of the minimum thickness and develops further into it, whereas the other one arrests for annihilation of the crack driving force (the other fracture has proceeded further and the slip line field that has caused the condition of instability has evaporated), with the evident crack tip blunting absorbing any kinetic energy of the non proceeding fracture. A portion of material is almost completely isolated. The zone of interference of the two shear fractures is a zone of transition from a pure mode-II fracture into the mode-I which is characteristic of the region of minimum thickness rupture. Examination of a polished section of the same specimen has allowed to establish that multiple ruptures concurrently occur, clearly detailing a slip line field (Fig. 6). When a ρ = 3.0 mm gas nitrided low alloy steel notched sample is examined, the same mechanism of fracture is evident, with the first cracks reaching a depth of about 120 μm (Fig. 7).
The same path holding for all gas, salt or plasma heat-treated samples, a complete rationalization of the fracture process in a complete model, valid for notched nitrided or nitrocarburized three-point bending samples, is presented with the aid of Fig. 8.

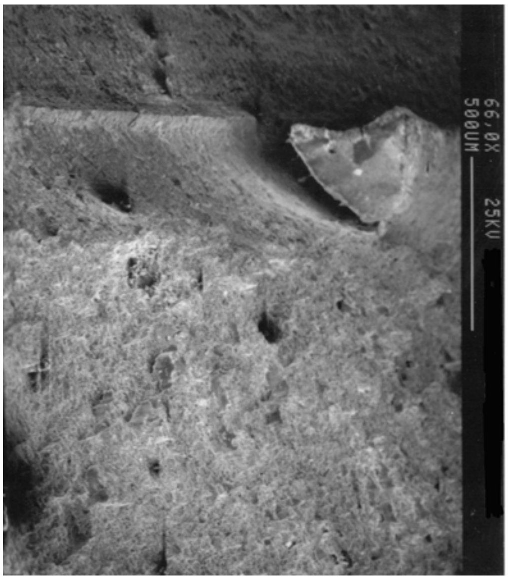

Figure 5 - Overall view of the tilted fracture surface of the broken half of a salt nitrocarburized AISI H-13 steel sample (Charpy-V notch, ρ = 0.25 mm).

Figure 6 – Polished cross section of the broken half of a salt nitrocarburized AISI H-13 steel sample (Charpy-V notch, ρ = 0.25 mm).

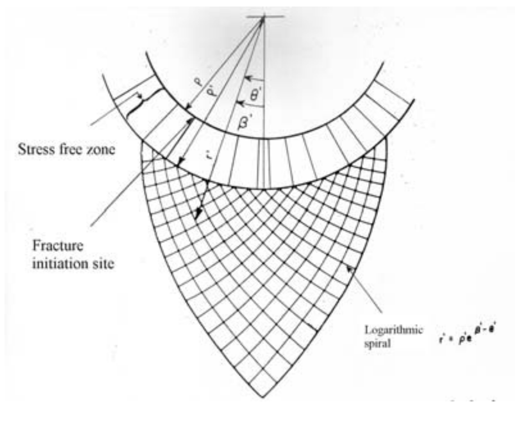

Figure 7. Overall view of the fracture surface of the broken half of a plasma nitrided low alloy steel sample (Charpy-V notch, ρ = 3.0 mm).

Fig. 8 - Stress free zone and slip line field forming at the root of the notch in an impact tested nitrided steel sample.

I. when the hammer strikes the sample surface opposite to the notch of radius ρ, multiple mode-I radial cracks develop at the root of the notch encompassing both the top thin nitride layer and most of the diffusion zone, with their length being in relation with the nitrogen diffusion depth but not coinciding with it (i.e. the larger the diffusion depth the longer the radial cracks);
II. a large stress-free zone thus develops at the root of the notch, with its outer perimeter copying the notch surface at a larger radius, ρ';
III. on such a nominal stress-free surface a slip line field develops, followed by the concurrent formation of multiple logarithmic spiral cracks competing each other to reach the region of minimum thickness; they are obviously mode-II fractures;
IV. the race restricts to two of them, with one hitting the notch centerline before the other and further propagating into the region of minimum thickness with a mode-I fracture. Definite large shear lips are thus formed in the fracture process.

3 CONCLUSIONS

After a review of the mechanical properties of nitrided and nitrocarburized workpieces giving raise to wear and high cycle fatigue resistance, attention has been devoted to fracture initiation mechanisms usually active in low cycle fatigue. By examining fracture surfaces of nitrided or nitrocarburized impact tested blunt notch specimens, a detailed sequence of steps yielding a definite shear lip formation by a partial mode-II fracture along a slip line field has been singled out.

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