ELSEVIER

Int. J. Fatigue Vol. 19, No. 1, pp. 67–73, 1997

0142–1123/97/$17.00

PII: S0142-1123(96)00031-X

Effect of nitriding on low-cycle fatigue properties

M. Guagliano and L. Vergani

Dipartimento di Meccanica, Politecnico di Milano, Piazza Leonardo da Vinci, 32, 20133 Milano, Italy
(Received 24 October 1995; revised 6 March 1996)

Nitriding is a thermochemical surface treatment that is often utilized to improve the fatigue strength of mechanical elements. In fact it is well known that, after the treatment, owing to the formation of a nitrided external layer and a field of residual stresses that are compressive on the surface, the high-cycle strength is strongly increased. Treatment characteristics include temperature and duration, whose values are generally decided by the operator. In this study, experimental axial tests were conducted on smooth specimen groups, which were each treated by using different durations. The results ιow the strong influence of treatment time on the elastic modulus values: in particular, if the duration increases, the elastic modulus increases too. A simple model allowed us to evaluate the distribution of the stresses in the case and in the core, and it was found that by increasing the treatment time the external layer is more stressed with respect to the core. Low-cycle fatigue was investigated, and the ϵ_a–N_f curves obtained by means of the differently treated specimens were compared. The results show that increasing the duration decreases the fatigue behaviour for high applied strains, and only one overload may be sufficient to fracture the external layer. Copyright © 1997 Elsevier Science Limited.

(Keywords: nitriding; treatment characteristics; low-cycle fatigue)

It is well known that thermochemical treatments improve fatigue strength in the high-cycle region[1–3], owing to the production of a case-hardened layer on the material surfaces. This external layer is characterized by high strength, a relatively brittle behaviour, and compressive residual stresses. In contrast, the core of the material is softer and rather ductile, while the residual stresses are lower and tensile, in order to balance the external ones.

Mechanical characteristic improvements depend upon a great variety of metallurgical and mechanical considerations. In fact, when thermochemical treatments such as nitriding and carburizing alter the chemical composition of the surface layer, the transformation kinetics and tempering responses are different in the regions of the resulting composite, so leading to a core and a case, with different microstructures and different responses to deformations.

In order to understand the overall mechanical behaviour two approaches are generally followed.

One is based on the net fatigue strength, which is determined by considering the strength increase due both to the residual stress state and to the hardness of the external layer. If the net strength pattern is compared with the applied stress it is possible to forecast where the cracks initiate. This concept is utilized when there is a stress gradient[2,4] in order to justify the position of the crack initiation: in fact the cracks start in the external layer when there are strong notches and in the subsurface region when the specimens are smooth or slightly notched. Macherauch *et al*[5,6] considered the same approach with the residual stresses as localized mean stresses, and Tan[7] gave a relationship that considers the residual stress effect for notches to determine the fatigue strength.

With the second approach, the surface-hardened component is considered as a composite consisting of a high-strength, low-ductility case and a lower-strength, higher-ductility core.

Landgraf[8] conducted experimental tests on smooth axial specimens with different case depths, due to different carburizing process parameters, to determine cyclic stress–strain and strain–life curves. The results show an improvement of the high-cycle fatigue strength (and subsurface cracks) if the carburized external layer depth increases, but in low-cycle fatigue the carburized steel is inferior to the uncarburized one (and the cracks are external), with the deepest case giving the shortest life.

Bäumel and Seeger[9] considered a model consisting of two cylinders, the case and the core, each characterized by different residual stress patterns and stress–strain curves. They conducted experimental axial tests by means of smooth nitrided specimens, and they always found a better behaviour of the treated specimens with respect to the untreated ones, in both low-cycle and high-cycle fatigue. They observed a shift of the location of the crack initiation from the surface to the core region, by decreasing the imposed values of strain. It is important to remember that the white layer was removed from the nitrided specimens.

Bacher *et al*[10] considered a similar multi-layer model to show the compressive residual longitudinal stress relaxation when a tension load is applied to a nitrided specimen; owing to the different transverse coefficient, the circumferential residual stress increases when the core yields, and the case remains linear-elastic. According to ref. 1, a variation in Poisson's ratio is the cause of the elongation reduction of the nitrided specimen: in fact, in the region between the layer and the case, the stress state is multiaxial, and this acts as a stress concentration factor.

Qian and Fatemi[1], using ion-nitrided specimens, showed that in the low-cycle region the strength of the nitrided specimens decreases by an order of magnitude with respect to the non-nitrided data, and the crack initiation shifts on the external surface. This is attributed to different factors. The larger strain amplitudes applied to the specimens relax the compressive residual stresses induced by the surface treatment: the case is characterized by a brittle behaviour and a low ductility, which reduces fatigue resistance in low-cycle fatigue, when plasticization is extensive.

In the present study the low-cycle fatigue behaviour of gas-nitrided steel (42CrMo4 EN10083) is considered. The nitriding treatment was carried out in two phases, characterized by different temperatures, durations and ammonium dissociation degrees. The temperature and duration strongly influence the hardness and the depth of the external case. In fact, by increasing the temperature, the surface peak hardness decreases, while the treatment diffusion becomes larger and the layer depth increases. The limit to the temperature increase is constituted by an excessive distortion of the element and by an excessive white layer depth. The duration increases the layer depth and the hardness pattern is also modified; initially, by increasing treatment duration, the surface becomes harder, but if a limit is exceeded the peak shifts towards the core and the surface hardness decreases.

Three different treatment times were used, and the stress–strain and strain–life curves were considered with a view to evaluating the influence of the treatment time.

A simple model based on the different stress–strain curves of the external layer and the core material is proposed to justify the different crack positions caused by the different treatment conditions and the different strain imposed values.

MATERIAL AND TREATMENTS

The chemical composition of the steel 42CrMo4 EN10083 is shown in *Table 1*. The material was quenched (850°C), cooled in oil and tempered (580°C).

Table 1 Chemical composition of 42CrMo4 (%)

C	Mn	Si	Cr	Mo	Al	Ni	S	P
0.39	0.81	0.31	1.13	0.18	0.012	–	0.020	0.022

In *Table 2* the mechanical and cyclic characteristics of the quenched and tempered material are shown. The monotonic parameters were experimentally determined, and the quenched and tempered cyclic ones were found from the literature[11].

Gas nitriding was executed by means of two phases and by imposing the parameters listed in *Table 3*. The total time necessary for treatment is 30 h. The treatment parameters were changed by varying both the temperature and the duration. In particular a lower total duration (10 h) was considered (*Table 4*) and a greater duration (70 h) (*Table 5*).

Numerous experimental tests were performed on the specimens treated.

At the end of the tests, the broken specimens were observed through an optical microscope to examine the fracture surface and to establish whether the crack initiation point was on the external surface or under it.

The white layer dimension was also measured. It was discovered that in the 10 h treatment this is completely absent, while in the 30 h treatment it has a value between 3 and 7 μm; in the 70 h treatment this value is between 7 and 14 μm.

The tests were executed without removing the white layer from the specimens, in order to simulate the working conditions of the mechanical components.

30 h gas-nitrided specimens

The mechanical characteristics were experimentally determined, and are reported in *Table 2*. In *Figure 1* the monotonic stress–strain curve is compared with the stress–strain curve of the untreated material.

The residual stresses were measured by means of an X-ray diffractometer. Numerous measurements were taken on the surface, and the average values were, in the longitudinal direction, $\sigma_{rl} = -320$ MPa ± 10% and, in the circumferential direction, $\sigma_{rc} = -150$ MPa ± 10%. The residual stress pattern is related to the hardness profile, which was found by means of microhardness measurements (*Figure 2*). The surface hardness is 520 HV, and the value below the surface increases gradually until the maximum (650 HV) is reached and then decreases to the core value, which is about 350 HV. It is possible to establish the nitrided layer dimension, resulting from the hardness profile, as about equal to 0.3 mm.

The cyclic properties of the treated specimens were experimentally determined by means of smooth specimens (diameter equal to 8 mm) and according to the ASTM E606-95 standard. The tests were carried out at room temperature under total strain control using a 100 kN Schenck-Trebel testing system.

10 h gas nitriding

The mechanical characteristics were experimentally determined, and are reported in *Table 2*. In *Figure 1* the monotonic stress–strain curve is compared with the curves of the other treatments.

The microhardness was measured, and *Figure 2* shows the hardness profile. Compared with the 30 h gas-nitriding layer the thickness of the nitrided layer proves to be lower, i.e. equal to about 0.2 mm, as is shown in *Figure 3*. The maximum hardness value (510 HV) is found on the external surface, and is lower than that of the 30 h treatment.

The cyclic characteristics were experimentally

Table 2 Mechanical and cyclic characteristics of the quenched and tempered 42CrMo4 and the nitrided 42CrMo4

	Mechanical characteristics			Cyclic characteristics[b]		Coffin–Manson parameters[c]			
	E (MPa)	$R_{p0.2}$ (MPa)	R_m (MPa)	n'	K' (MPa)	b	c	σ'_f (MPa)	ϵ'_f (m/m)
42CrMo4	201.000	813	1088	0.087	807	−0.091	−0.837	1036	2.251
30 h	208.000	1046	1240	0.247	3780	−0.057	−0.225	1330	0.012
10 h	203.000	963	1045	0.106	1413	−0.058	−0.548	1148	0.110
70 h	235.000	930[a]	960[a]	–	–	−0.070	−0.787	1213	0.073

[a]Values determined by considering the initial resistant section. If the core section is considered the following values are found: $R_{p0.2}$ = 1150 MPa, R_m = 1185 MPa
[b]$\epsilon_a = \sigma_a/E + (\sigma_a/K')^{1/n'}$
[c]$\epsilon_a = (\sigma'_f/E)\,(2N_f)^b + \epsilon'_f\,(2N_f)^c$

Table 3 30 h treatment

	Temperature (°C)	Duration (h)	Dissociation degree [%]
Phase I	510	10	20
Phase II	530	20	40–65

Table 4 10 h treatment

	Temperature (°C)	Duration (h)	Dissociation degree [%]
Phase I	505	5	20
Phase II	525	5	50–70

Table 5 70 h treatment

	Temperature (°C)	Duration (h)	Dissociation degree [%]
Phase I	510	15	20
Phase II	525	55	50–70

determined by following the same procedure mentioned above.

The surface residual stresses were measured too, by means of an X-ray diffractometer, and the longitudinal value was found to be equal to σ_{rl} = −430 MPa ± 10%.

70 h gas nitriding

Figure 1 shows the stress–strain curve obtained by means of a tensile test. It displays a discontinuity corresponding to a low strain value (about 2000 μm m^{-1}) due to the fracture of the external nitrided layer. *Figure 4* shows a helicoidal fracture along all the specimen, inclined at a slight angle. After the case fracture the test continued until the core strength was reached. The curve reported in *Figure 1* was obtained by considering the specimen total section, even if after the fracture of the external layer the resistant section was reduced.

Figure 2 also shows the hardness profile of the 70 h nitrided specimens. The surface value is equal to 540 HV. The maximum hardness value (650 HV) is

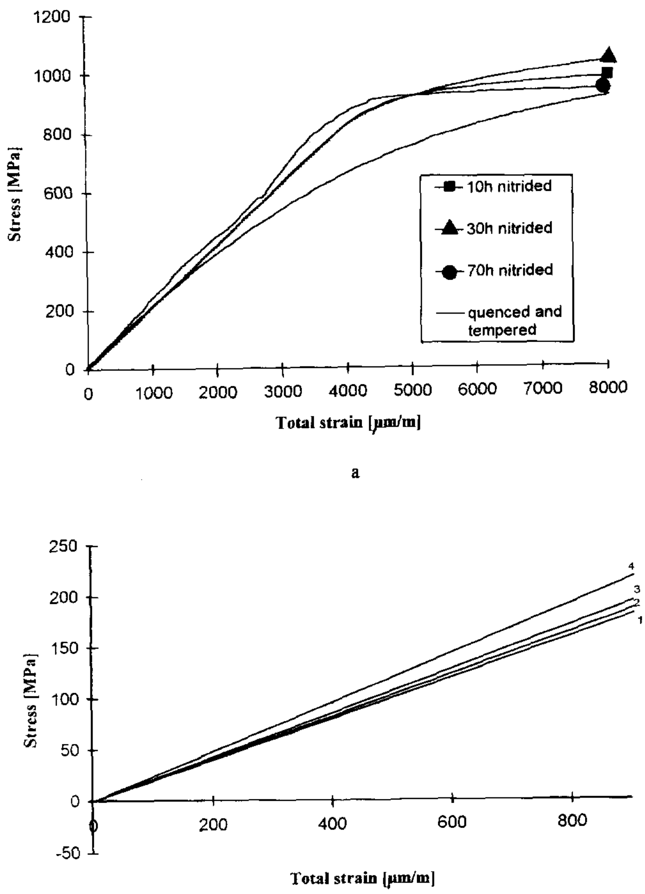

Figure 1 Quenched and tempered and 10 h, 30 h and 70 h nitrided 42CrMo4: (a) stress–strain curves; (b) details of the linear–elastic field: (1) quenched and tempered 42CrMo4, (2) 10 h nitrided, (3) 30 h nitrided, (4) 70 h nitrided

shifted towards the inside, and it is lower than for the 30 h treatment; furthermore, the nitrided layer dimension is increased up to about 0.4 mm.

It is evident that this trend is not significantly different with respect to the 30 h HV profile. However, from the pictures shown in *Figure 5*, it is possible to note the different depths of material hardened by the treatments. In addition, the mechanical and cyclic characteristics of these two groups of specimens are very different, and it is necessary to distinguish between the differently treated specimens.

The surface residual stresses were measured too, and

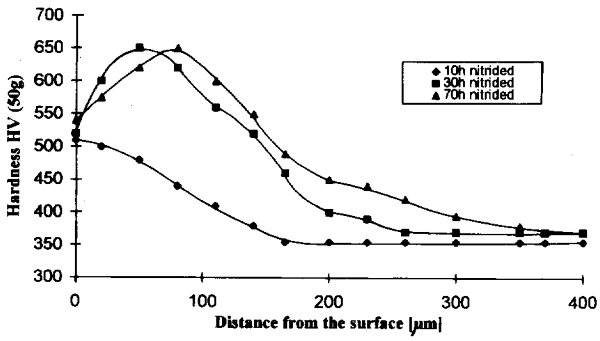

Figure 2 Hardness profiles

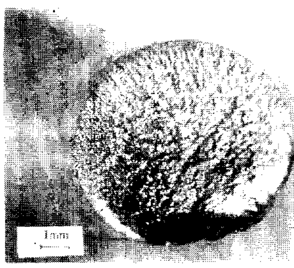

Figure 3 Fracture section of 10 h nitrided specimen: it is possible to see the external layer

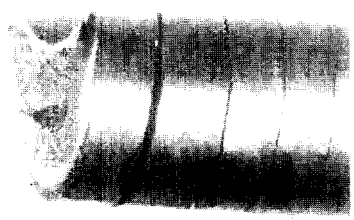

Figure 4 Photograph of a 70 h nitrided specimen fracture section after the tensile test: it is possible to see the helicoidal fracture of the external case

the values found were completely different from the previous results, in fact σ_{rl} = 90 MPa ± 10%, probably due to the presence of the white layer.

The cyclic characteristics were experimentally determined by following the same ASTM standard.

DISCUSSION OF THE RESULTS

The first thing that appears clear is that by increasing the treatment time the depth of the nitrided layer increases, and the global stiffness of the specimen increases too. In fact the elastic longitudinal modulus E passes from 201.000 MPa for the quenched and tempered material to 203.000 MPa for the 10 h nitriding, to 208.000 MPa for the 30 h nitriding, and to 235.000 MPa for the 70 h nitriding. Assuming that the elastic modulus of the core is always equal to the quenched and tempered one, as the elongation is the same on all the specimen sections, the stress in the nitrided layer is different from the stress in the core, owing to the different elastic moduli. If these are considered constant in the nitrided layer and the core, it is possible to determine an approximate value of the elastic modulus of the nitrided layer by means of a simple model of two springs connected in parallel, which is shown in *Figure 6*.

By balancing the forces, it is possible to write

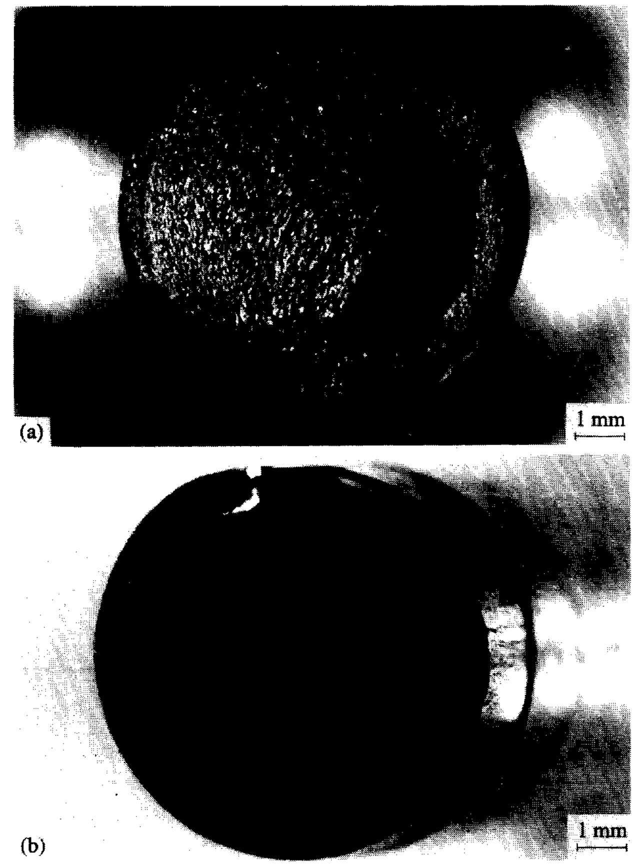

Figure 5 (a) Photograph of a 30 h specimen fracture section; (b) photograph of a 70 h specimen fracture section

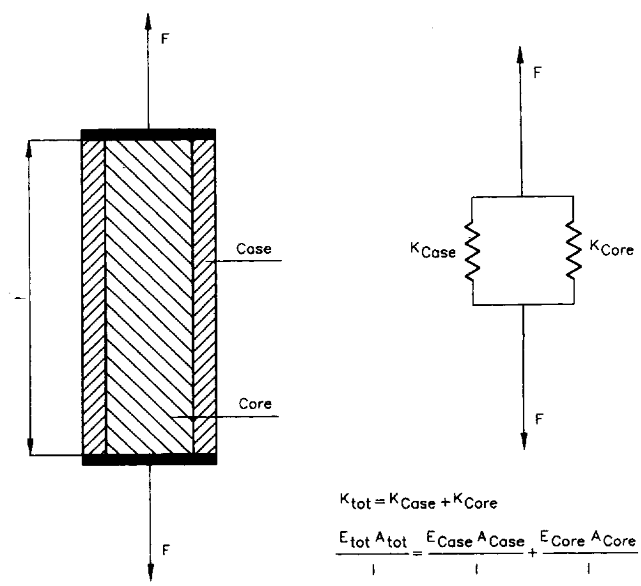

Figure 6 Scheme of the simplified model

$$E_{case} = \frac{E_{tot}A_{tot} - E_{core}A_{core}}{A_{case}}$$

The values obtained utilizing this equation are:

10 h nitrided layer	E = 221.000 MPa
30 h nitrided layer	E = 249.000 MPa
70 h nitrided layer	E = 380.000 MPa

Table 6 Low-cycle fatigue results

	10 h nitrided		30 h nitrided		70 h nitrided	
ϵ_a (μm m^{-1})	N_f	Crack	N_f	Crack	N_f	Crack
±2000	–	–	–	–	10^6 cycles unbroken	–
±2500	–	–	–	–	8000	External
±3000	–	–	–	–	650	External
±4000	–	–	33 123	Internal	180	External
±5000	956	Internal	–	–	–	–
±6000	711	Internal	4 171	Internal	80	External
±6500	–	–	1 430	Internal	–	–
±7000	371	Internal	337	External	30	External
±8000	273	External	100	External	–	–
±9000	138	External	50	External	–	–
±10000	90	External	20	External	–	–

This is a simplified model of what actually happens: in fact the elastic modulus values are probably not uniform on the external layer and on the core. However, this simple model justifies the apparently anomalous behaviour of the 70 h nitrided specimen. In fact, by considering this elastic modulus, even if the strain value is low, the stress supported by the case is very high, and it may reach the tensile strength.

The helicoidal fracture could be justified by considering a multiaxial stress state due to the different behaviours of the case and the core, also observed in refs 12 and 13.

If the residual stress values are considered, this phenomenon is amplified for the 70 h treatment specimens, where tensile residual stresses were measured. However, for the other treatment durations, the compressive residual stresses attenuate the effect of the different elastic moduli of the case and the core.

The fatigue test results (corresponding to complete fracture of the specimens) are included in *Table 6* and shown in *Figure 7*. It is possible to note the different behaviour of the nitrided specimens. It is also evident that by increasing the strain amplitude the results are not favourable, and this trend becomes worse if the treatment duration increases.

Even the crack initiation position is strongly influenced by the duration of the nitriding treatment and the strain value applied. In fact the crack begins to propagate under the nitrided layer if the stress amplitude is low. In contrast, in the case of 70 h treatment, all the specimens show external cracks, except for a specimen that remained unbroken after 2×10^6 cycles.

In *Figure 8* an internal crack beginning from an inclusion, and an external crack beginning on the surface, are shown. The internal cracks always start from an inclusion, according to data reported in refs 1–3.

The mechanical model described above has been used to explain these results too. From the data of *Table 6* the corresponding cyclic curves are determined

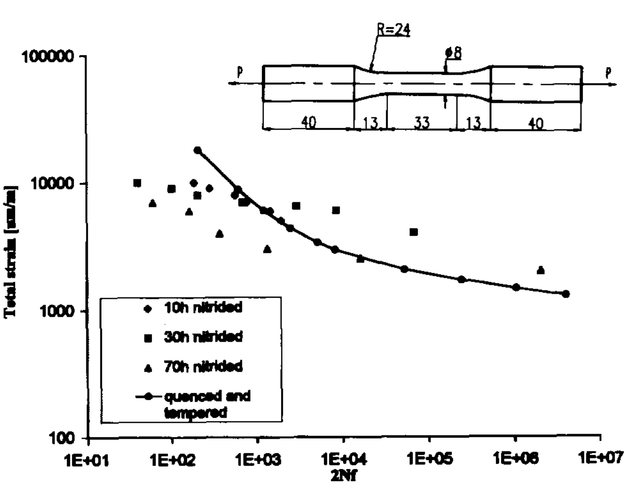

Figure 7 Low-cycle fatigue results of the un-nitrided and nitrided specimens and scheme of the specimens utilized

Figure 8 (a) Internal crack, beginning from an inclusion; (b) external crack, beginning on the surface

and shown in *Figure 9*. The 70 h treatment cyclic curve is not reported because these experimental data are not homogeneous with the other, owing to the fracture of the nitrided layer during the first loading cycle, corresponding to a strain about equal to 2500 μm m^{-1}.

From the figure it is clear that by increasing the treatment time the cyclic hardening increases. If the applied strain is high the result is that the external layer is more stressed than the core, owing to the different values of the elastic moduli to the yielding of the core, which has a ductile behaviour.

If lower strain amplitudes are considered, it can be noted that the 30 h and 70 h nitrided axial fatigue strength improves; in order to explain this trend the previous simplified model can be recalled. In fact the stress profile over the section is not uniform, and the core is less stressed than the quenched and tempered specimens. If this overloading of the external layer is not excessive, the resulting strength improves.

The 10 h treatment specimens show a shorter life with respect to the un-nitrided specimens, even if the cracks initiate internally. This could be justified by considering the large compressive residual stresses measured on the surface, which cause high tensile residual stresses in the core. In addition, the elastic modulus of the case is similar to that of the core: consequently the global stresses are not sufficiently diminished in the core and the axial fatigue behaviour is not improved.

Another parameter that seems to be characteristic of the nitrided specimens fatigue behaviour is the transition number of cycles, N_t: that is, the number of cycles when the elastic strain amplitude equals the plastic one. It is evaluated from the following expression:

$$N_t = \frac{1}{2}\left(\frac{\sigma_f'}{\epsilon_f' E}\right)^{\frac{1}{c-b}}$$

This is shown in *Figure 10* with respect to the nitriding time, and it seems to follow the same trend as the low-cycle fatigue behaviour; in fact N_t reduces for greater treatment time.

CONCLUSIONS

Experimental tests were carried out on cylindrical smooth specimens subjected to different nitriding times. In particular, the low-cycle fatigue range was investigated, and the following conclusions can be drawn.

1. Three different durations were considered: 10 h, 30 h and 70 h. By increasing the treatment time, deeper nitrided layers are obtained. The elastic modulus is strongly influenced by the different treatments, and the stress–strain curves are consequently different.
2. A simple and approximate model was developed, by considering the case and core as springs with different stiffness connected in parallel. In this way it is possible to evaluate approximately the actual stress values in the core and in the case. As the stiffness values are different the stress profile is not uniform, and in particular the case is more stressed than the core. The stiffness of the external layer increases with the treatment time, and consequently the overstress increases too. Therefore the 70 h nitrided layer fractures in correspondence to a low nominal stress value.
3. Low-cycle axial fatigue tests were carried out on differently treated smooth specimens. For the lowest strain values the cracks initiate in the core, corresponding to an internal inclusion; in contrast, for the highest strain values the cracks start on the external surface.

   If these fatigue results are compared with the ones obtained for quenched and tempered specimens it is possible to see that when the cracks begin internally there is an improvement, while if the cracks are external there is a worsening of the fatigue strength. In practical applications, attention must be paid to the choice of the treatment time, because if the treatment time is long, few overloads are sufficient to fracture the hardened layer and to start a fatigue crack.
4. The model described in conclusion 2 was utilized to explain the low- and high-fatigue results. In this way it is possible to understand not only the behaviour of the nitrided specimens at the highest strain amplitudes, but also the improvement of the axial fatigue strength for the lowest imposed strains (30 h and 70 h specimens). In fact, in this case the stresses in the external layer are not dangerous, and the stresses in the core are lower than the nominal ones. The model also justifies the worse behaviour of the 10 h treated specimens; in fact in this instance the differences between the elastic modulus values of the case and the core are not enough to improve the fatigue resistance, and the very high compressive residual stresses on the surface are balanced by

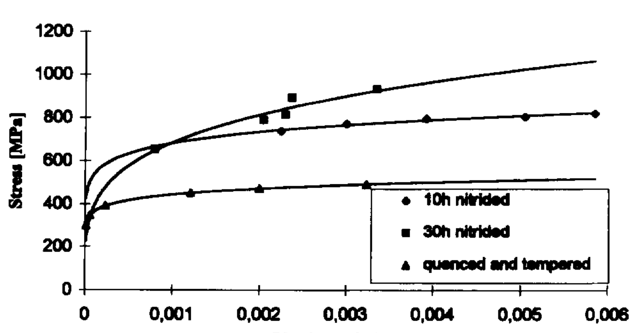

Figure 9 Cyclic curves

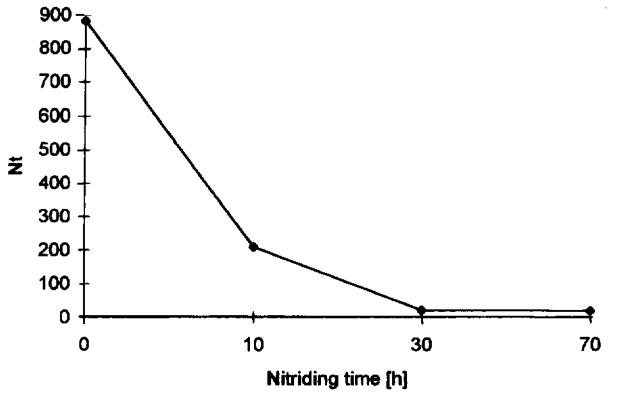

Figure 10 Transition number of cycles in relation to the treatment time

internal tensile stresses, which are not favourable to the fatigue behaviour.

5. The transition number of cycles, N_t, reduces if the treatment time increases. This fact can be considered as an index of the lower ductility obtained by increasing the depth of the nitrided layer and, consequently, a cause of the worst behaviour reached for high strain amplitudes by using long nitriding time.

ACKNOWLEDGEMENTS

We should like to thank our colleague Marco Boniardi for his help during the development of this paper. This work has been supported by a MURST grant.

REFERENCES

1 Qian, J. and Fatemi, A. *Int. J. Fatigue* 1995, **17** (1), 15
2 Spies, H.J. Fatigue behaviour of nitrided steels. *Steel Research*, 1993, **64** (8/9), 441
3 Balbi, M., Boniardi, M., Guagliano, M. and Vergani, L. In 'Atti IX Convegno Nazionale Gruppo Italiano Frattura (IGF9)', Rome, 1993, pp. 161–170
4 Woodvine, J.G.R. Carnegie Scholarship Mem. 13, Iron Steel Inst., 1924, pp. 197–237
5 Macherauch, E. and Kloos, K.H. In 'Eigenspannungenund Lastspannungen', Härterei-Tech., Mitt. Beiheft, 1982, pp. 175–194
6 Kloos, K.H. and Velten, E. *Konstruktion* 1984, **36** (5), 181
7 Tan, N.D. Untersuchung des Zusammenhanges zwischen dem Aufbau von Nitrierschichten und der Schwingsfestigkeit gekerbter Proben, (Dr -Ing. Diss. Bergakademie Freiberg), 1990
8 Landgraf, R.W. and Richman, R.H. In 'Fatigue of Composites Materials'. ASTM STP 569, American Society for Testing and Materials, Philadelphia, 1975, pp. 130–144
9 Baumel, A. Jr and Seeger, T. In 'Proc. II International Conference on Residual Stresses (ICRS2)', Nancy, 1988, pp. 809–814
10 Bacher, M., Zhang, D., Scholtes, B., Vöhringer and Macherauch, E. In 'Proc. II International Conference on Residual Stresses (ICRS2)', Nancy, 1988, pp. 753–758
11 Boller, Chr. and Seeger, T. 'Materials Data for Cyclic Loading', Elsevier Science Publishers, Amsterdam, 1987, p. 482
12 Magnusson, L. 'Cyclic Behaviour of Carburized Steel', Linkoping Studies in Science and Technology, No. 56, Linkoping University, Sweden, 1980
13 Ebert, L.J. *Metall. Trans.* 1978, **9A**, 1537