

EFFECT OF PLASMA NITRIDING ON CYCLIC BEHAVIOUR UNDER VARIOUS ENVIRONMENTAL CONDITIONS

K.-T. Rie, Th. Lampe and W. Kohler \*

The influence of plasma nitriding on fatigue behaviour of steels has been investigated in air, tap water, salt water and gaseous hydrogen. Tests were carried out in high- and low-cycle fatigue regime. Both tension-compression and cyclic torsion were chosen as the mode of force application. It has been found that the plasma nitriding improves the fatigue strength in air as well as in corrosive environment. The reasons for this improvement were discussed in light of the microstructural features and the fracture morphology.

INTRODUCTION

Plasma nitriding is now one of the most up-to-date surface treatment methods. Previous work has shown that the microstructure of the surface layer (1), (2) and the torsional fatigue strength can be affected (3) by plasma nitriding. The effect of plasma nitriding on fatigue strength has been studied mostly in high-cycle fatigue regime in air.

Therefore the object of this study was to investigate the influence of microstructure on fatigue behaviour in high-cycle as well as in low-cycle fatigue regime. Various environmental conditions were included in the study.

EXPERIMENTAL PROCEDURE

The low alloyed steels 34 CrAlMo 5 (quenched and tempered) and 25 CrMo 4 (annealed) were selected for study. Plasma nitriding was performed at 560° C for 3 hours in two different N<sub>2</sub>/H<sub>2</sub>-mixtures, producing nitrided cases with various microstructure. After nitriding in 80 % N<sub>2</sub>/20 % H<sub>2</sub> mixture a polyphase compound layer (white layer in Fig. 1) consisting of  $\gamma'$ -Fe<sub>4</sub>N and  $\epsilon$ -Fe<sub>2-3</sub>N is formed. In 20 % N<sub>2</sub>/80 % H<sub>2</sub> mixture a monophase  $\gamma'$ -Fe<sub>4</sub>N compound layer arises. The dimensions of nitrided cases are given in Table 1.

\* Institut für Schweißtechnik und Werkstofftechnologie, TU Braunschweig

TABLE 1 - Dimensions of Nitrided Cases

Material	Gas Mixture	Compound Layer	Effective Nitrided Case Depth
34 CrAlMo 5	80 % N <sub>2</sub> /20 % H <sub>2</sub>	10 - 12 μm	0,25 mm
	20 % N <sub>2</sub> /80 % H <sub>2</sub>	2 - 4 μm	
25 CrMo 4	80 % N <sub>2</sub> /20 % H <sub>2</sub>	13 - 15 μm	0,28 mm

Cyclic torsion and tension-compression were used as the mode of force application in high-cycle fatigue (HCF) regime. In low-cycle fatigue (LCF) regime tests were carried out in strain controlled tension-compression. All tests were conducted in various environments such as air, tap water, artificial sea water (DIN 50 905) and high pressure hydrogen of 10 MPa. The criterion used for fatigue failure in HCF-regime was the number of cycles to fracture  $N_f$  and in LCF-regime the critical number of cycles  $N_{cr}$  (4). The results of the fractographic investigations were related to the crack initiation and propagation mechanisms.

#### RESULTS AND DISCUSSION

Plasma nitriding leads to an increase of the resistance to fatigue of metals. In cyclic torsion the fatigue limit of 34 CrAlMo 5 increases about 40 % due to plasma nitriding (Fig. 2). The fatigue limit of the specimen is not influenced by the microstructure of the compound layer. In cyclic stressing above the fatigue limit the fatigue strength of specimen with polyphase  $\epsilon + \gamma'$  compound layer is slightly higher than that with monophase  $\gamma'$ . SEM investigation of the fracture surface has revealed that the crack initiates at the surface, when stressed above the fatigue limit, while cyclic stressing around fatigue limit produces subsurface cracks. The influence of compound layer structure on the fatigue behaviour is related to the different hardness of the compound layers. The fatigue strength above the fatigue limit increases with increasing hardness of the compound layer. This results are comparable with those by Bahre (5).

For tension-compression an increase of the fatigue limit by 20 % was observed for 25 CrMo 4 (Fig. 3) after plasma nitriding. The crack initiation was found in un-nitrided core, when tested in region of  $N > 10^4$  cycles (Fig. 5). Most of them starts from an inclusion composed of aluminium (Fig. 6).

The results of LCF-testing with constant strain ranges (Fig. 11a) show that the plasma nitriding leads to an improvement of fatigue life for strain ranges up to 1,0 %. The subsurface crack initiation in this region is comparable with that in stress controlled HCF-tests. In the region of high strain ranges ( $\Delta\epsilon_a > 1,0$  %) the crack initiation occurs at the surface leading to the observed reduction of the fatigue life. No unique slope of the fatigue life curve was found. The transition strain range is around  $\Delta\epsilon_a \approx 1,2$  %. The shape of the fatigue life curve does not depend on the microstructure of the compound layer.

Fig. 4 shows the cyclic strain hardening curves for plasma-nitrided specimens. By straining with strain range of  $\Delta\epsilon_a = 1,6$  % cracks formed after first few cycles. Therefore, the fatigue life was consumed mainly by crack

propagation. The corresponding cyclic flow curve shows a continuous cyclic softening character. At  $\Delta\epsilon_a = 1,2\%$  cracks started from the surface as observed at higher strain range; however the number of cycles to crack initiation was remarkably extended. The cyclic flow curve shows the corresponding characteristic shape: after initial cyclic strain softening a saturation stress level is reached, before a macro-crack formation occurs resulting in a rapid stress drop. The fractographic investigation has shown that the fracture mode in LCF-regime depends on the microstructure of the specimen; quasi-cleavage fracture has developed in the nitrogen enriched surface layer (Fig. 7), while ductile striations were found in the core (Fig. 8).

In HCF-regime as well as in LCF-regime, when relatively low strain amplitudes are applied, the fatigue behaviour of plasma-nitrided steels is characterized by a significant increase of fatigue life. This improvement is due to the subsurface crack initiation. Nitrogen enrichment by plasma nitriding results in a considerable increase of yield stress and Young's modulus (6) (7). The cyclic plastic deformation is restricted therefore in the interior of the specimen beneath the case; this could be demonstrated by TEM investigation. Characteristic microstructure for cyclic plastic deformation like cell structure was found only in the core of the nitrided specimen while the diffusion layer had no visible deformation structures.

At high strain amplitude the applied stress exceeds the fracture stress of the nitrided case prior to gross deformation. The nitrided layer breaks soon after few cycles in a brittle manner, so that the crack initiation stage is extremely short. The fatigue life is spent mostly by the crack propagation (Fig. 4) and in such case the plasma nitriding does not improve the fatigue life.

As shown in Fig. 9 and 10 the corrosion fatigue behaviour of plasma-nitrided 34CrAlMo 5 in cyclic torsion is improved compared with that of un-nitrided specimen. The fatigue limit of un-nitrided steel is reduced in tap water by 40% (limiting cycle  $N = 10^7$ ), while the torsional fatigue limit of the plasma-nitrided specimen is not influenced by tap water (Fig. 9). Fractographic investigation has shown that the crack nucleation of the plasma-nitrided specimen occurred beneath the case even in tap water. It has been observed (Fig. 10) that the torsional fatigue strength of plasma-nitrided as well as un-nitrided specimen decreases in artificial sea water (DIN 50 905). As expected, there appears no fatigue limit up to  $N = 10^7$  cycles. Nevertheless by plasma-nitriding the fatigue strength at  $N = 10^7$  increases by 60%. The  $(\epsilon + \gamma')$ -compound layer is thicker than the  $\gamma'$ -compound layer and shows a slightly better corrosion fatigue behaviour than the  $\gamma'$ -compound layer. SEM examination has shown that in artificial sea water the crack initiation for plasma-nitrided specimens occurs at the surface. A weak corrosion attack was found around the nucleus at the surface while the crack propagation beneath the case was accompanied with a severe corrosion. The cracks extended mainly intercrystalline in the nitrided case as well as in the core (8).

The effect of high pressure hydrogen on the fatigue behaviour of plasma-nitrided 25CrMo 4 in LCF-regime is given in Fig. 11b. In the region of small strain range ( $\Delta\epsilon_a < 1,0\%$ ) the fatigue properties are not influenced by high pressure hydrogen. The crack nucleation is located beneath the case. At high strain range the compound layer breaks brittle soon after few cycles regardless of the microstructure. A local hydrogen embrittlement occurs at this crack tip and the crack propagates extremely fast resulting in the decrease of the fatigue life.

The increase of the fatigue strength in corrosive medium is the result of the improved corrosion resistance of the plasma-nitrided case. These observations agree well with those by Butenko et al. (9). As soon as the compound layer breaks and loses the protective properties against corrosive medium, a local concentration of corrosion attack at the crack tip occurs and this leads to a faster propagation of the crack into the interior and results in final fracture.

#### CONCLUSIONS

It has been found that the plasma nitriding improves the fatigue behaviour in HCF- as well as in LCF-regime. Even in corrosive environment a remarkable improvement may be achieved. Microstructural changes caused by plasma nitriding is responsible for this behaviour.

#### SYMBOLS USED

- a = crack length (mm)  
 N = number of cycles  
 $N_{cr}$  = critical number of cycles  
 $N_f$  = number of cycles to failure  
 $\Delta \epsilon_a$  = total strain range  
 $\sigma_t$  = stress (N/mm<sup>2</sup>)

#### REFERENCES

1. Lampe, Th., and Rie, K.-T., 1981, Z. Metallkunde, 72, 169.
2. Rie, K.-T., and Lampe, Th., 1982, Z. Metallkunde, 73, 349.
3. Martin, J.W., and Gowda, C.V.B., 1973, Third Intern. Conf. Fracture, München.
4. Rie, K.-T., and Stüwe, H.P., 1973, Z. Metallkunde, 64, 37.
5. Bahre, K., 1977, Dissertation, TH Darmstadt.
6. Bierwirth, G., 1962, Dissertation, TH Darmstadt.
7. Kwak, B.M., and Kim, J.A., Private communication, KAIST, Seoul.
8. Rie, K.-T., and Lampe, Th., to be published in Werkstoffe und Korrosion
9. Butenko, O.I. et al, 1972, Fiz. Khim Mekhan. Mat., 8, 88.

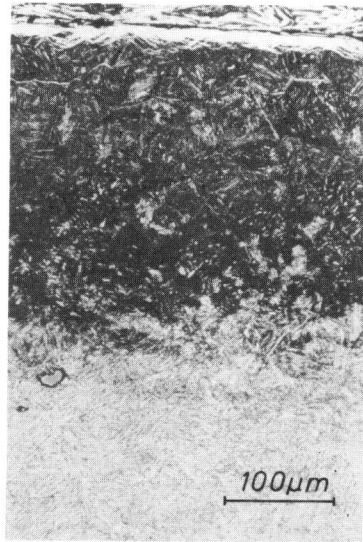


Figure 1 Plasma-nitrided layer of 34 CrAlMo 5

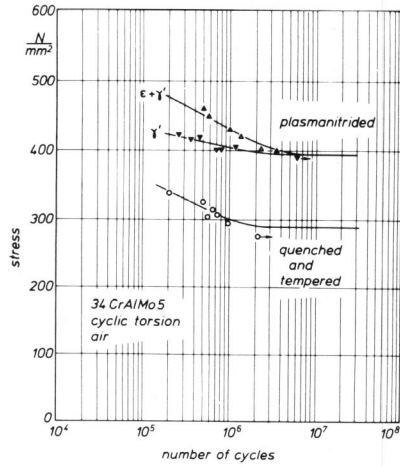


Figure 2 Influence of plasma nitriding on fatigue life in air

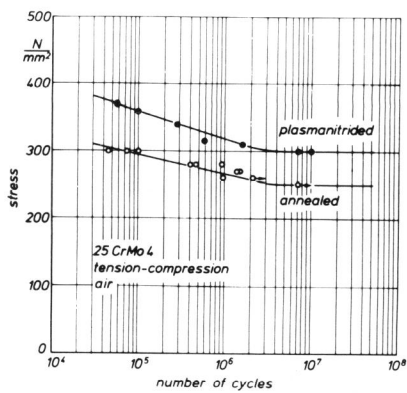


Figure 3 Influence of plasma nitriding on fatigue life in air

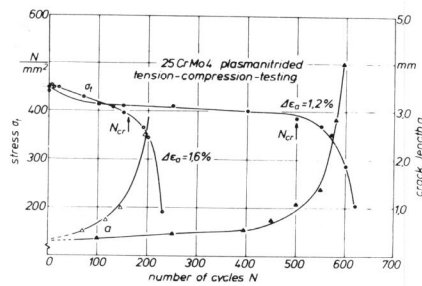


Figure 4 Cyclic flow curve and crack propagation for 25 CrMo 4

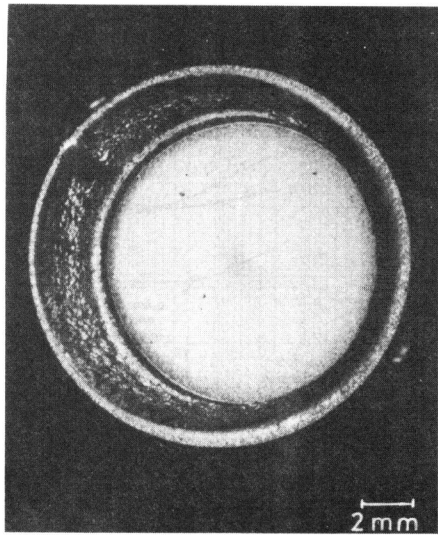


Figure 5 Fracture surface of plasma-nitrided tension-compression specimen

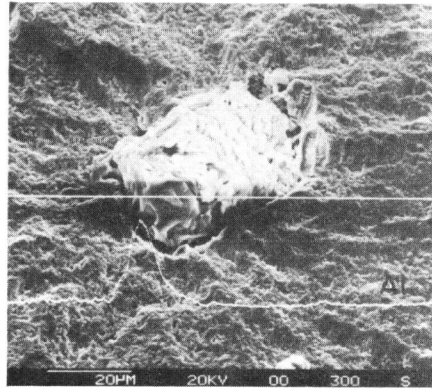


Figure 6 Aluminium-content inclusion on the fracture surface

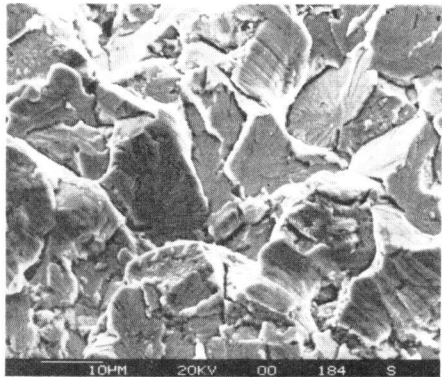


Figure 7 Quasi-cleavage fracture in the nitrided case of 25 CrMo 4

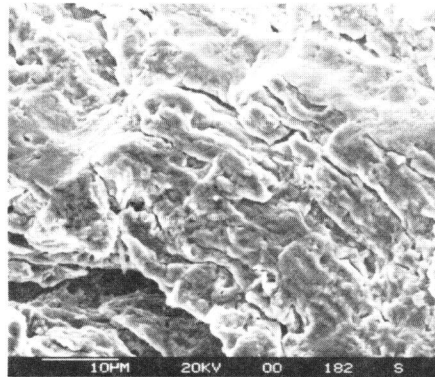


Figure 8 Ductile striations in the core of plasma-nitrided 25 CrMo 4

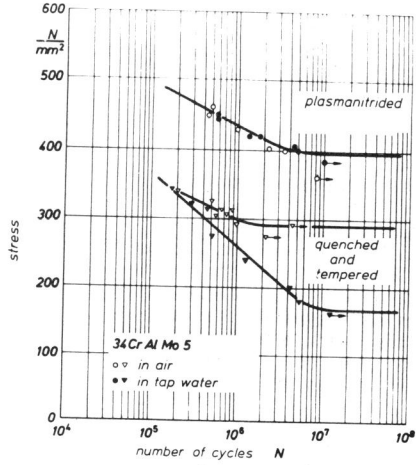


Figure 9 Fatigue life of plasma-nitrided 34 CrAlMo 5 in tap water

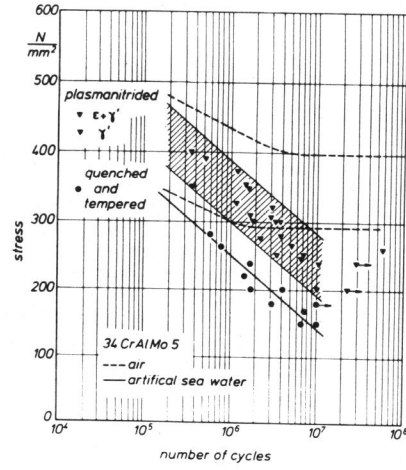


Figure 10 Fatigue life of plasma-nitrided 34 CrAlMo 5 in artificial sea water

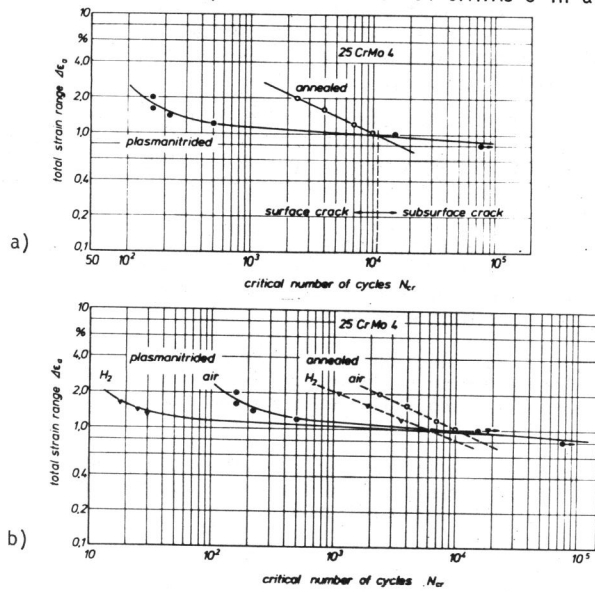


Figure 11 Fatigue life curves for plasmanitrided 25 CrMo 4 in air and in hydrogen environment