Fatigue Behaviour of New Duplex Stainless Steels Upgraded by Nitrogen Alloying

K. MASSOL, J.-B. VOGT and J. FOCT

Université des Sciences et Technologies de Lille, Laboratoire de Métallurgie Physique et Génie des Matériaux UMR CNRS 8517, Bâtiment C6, 59655 Villeneuve d’Ascq Cedex France. E-mail: jean-bernard.vogt@univ-lille1.fr

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The fatigue behaviour of two duplex stainless steels (DSS) alloyed with different nitrogen contents (0.24 and 0.4 wt%) has been studied in air and in a NaCl-containing water solution. Both of the alloys exhibit a cyclic softening preceded by a cyclic hardening at high strain amplitude as reported in the literature for lower nitrogen grades. In air, the fatigue lives are nearly the same for both of the DSS for any given strain amplitude. In the corrosive environment, the fatigue life was systematically reduced by a factor two for the 0.24% N-containing DSS in the strain range studied in contrast with the 0.4% N-containing DSS which appeared to be all the more insensitive as long as the strain amplitude was small. The fatigue resistance and the cyclic accommodation of these DSS are strongly controlled by the volume fraction of $\alpha$ and $\gamma$ phases. It is shown that DSS with a high fraction of austenite present a good combination of fatigue resistance and cyclic softening. Alloying with nitrogen appears to be a promising way to master an optimised microstructure leading to high mechanical resistant DSS.

KEYWORDS: low cycle fatigue; duplex stainless steels; high nitrogen steels; corrosion fatigue.

1. Introduction

More and more studies have been carried out on duplex stainless steels (DSS) due to their good mechanical properties associated with good corrosion resistance.1) These properties are essentially conditioned by the DSS microstructure, constituted by a ferritic matrix containing austenite islands, and by the thermal history (heat treatment, elaborating process . . .). Therefore the microstructure presents morphologic and sometimes crystallographic textures which are strongly dependent on the rolling process.2) The improved properties resulting from nitrogen addition on austenitic steels3) led us to examine the effect of nitrogen on DSS.

The purpose of this paper is a study of the fatigue behaviour of DSS alloyed with large contents of nitrogen. Attention will be paid to the role of the microstructure on the stress response to strain cycling especially during the accommodation period. It is also aimed to determine to which extent the fatigue resistance of DSS can be improved and which mechanisms are involved.

2. Experimental

2.1. Materials

Two different duplex stainless steels (see Fig. 1 for microstructures), produced at an industrial scale, and of which the compositions are reported in Table 1 were studied. The first one, produced by Usinor Industeel, is of the SAF 2507 type (labelled DSS–0.24%N) and contains 0.24 wt% of nitrogen. A solution heat treatment at 1 373 K for 3 000 s followed by a water quench results in a two phases microstructure constituted of 50% of ferrite and 50% of austenite. Due to the final dimensions obtained after rolling (sheet of 300 × 25 mm), the austenite islands are elongated in the rolling direction (more than 100 μm) and rather flat in the perpendicular one (around 10 × 30 μm). The microhardness is nearly the same for both of the phases, Hv10 = 310.

The second duplex stainless steel (labelled DSS–0.4%N) contains 0.4 wt% of nitrogen and a higher amount of Mn and W than the former one. This large nitrogen content, acting as a $\gamma$-stabiliser element, after an annealing treatment at 1 373 K for 1 600 s and water quenching led to a reduction of the volume fraction of ferrite to 30%. The nitrogen addition slightly hardens the steel resulting from a slight increase of the microhardness of the ferrite (Hv10 = 344) but especially that of the austenite (Hv10 = 363). At last, the elaboration as a rod material results in elongated and rounded austenitic islands ($\phi$= 25 μm).

2.2. Fatigue Tests

Low cycle fatigue tests have been performed on a servo-hydraulic machine used in a fully push-pull mode ($Re$ = −1). Cylindrical specimens, taken in the rolling direction (austenite fibres in the same way than the solicitation axis), had a reduced section of 6 mm and a gage length of 10 mm. Before testing, the specimen surface has been electro-polished using a solution containing 25% of acetic acid, 10% of perchloric acid and 1% of water. Tests have been carried out at room temperature and in the laboratory atmosphere.
(air), using a triangular wave form mode at a constant strain rate of $4 \cdot 10^{-3}$ s$^{-1}$. The deformation was measured by means of an extensometer directly stuck on the gauge length within a total strain range $\Delta e_t$ of 0.8 to 2%. The fatigue life $N_f$ is defined as the number of cycles up to a drop of 25% of the stress amplitude in relation with the stabilised stress amplitude ($\sigma_a$). The softening occurring while cycling is characterised by the softening rate $d(\Delta e_p)$ calculated from $\frac{\Delta e_p}{\Delta e_t}$.

### 3. Experimental Results

#### 3.1. Fatigue Behaviour of the Medium Nitrogen Containing DSS

The cyclic accommodation curves of the DSS–0.24%N (Fig. 2(a)) correspond to a behaviour which appears characteristic of duplex stainless steels. Two regimes of deformation have been clearly distinguished. At low strain level, the material softens immediately after the first cycle and then stabilises. At higher strain amplitudes, the material first starts hardening before softening and stabilising.

The fatigue resistance curves (also called Coffin–Manson curves, Fig. 2(b)) shows a slope discontinuity of the total and plastic variations which is noticeable for $\Delta e_t=1.3\%$. The corresponding Eqs. (1) and (2) for the plastic strain variation are the following:

$$\Delta e_p=48 \cdot N_f^{-0.53} \quad \text{for } \Delta e_t<1.3\% \quad \text{(1)}$$

$$\Delta e_p=7 \cdot N_f^{-0.27} \quad \text{for } \Delta e_t>1.3\% \quad \text{(2)}$$

As proposed by Magnin et al., the fatigue behaviour of this two-phase material refers to the response of a purely ferritic steel at high strain amplitude and to an austenitic one at low strain amplitude. These results from the macroscopic mechanical response have been associated with SEM observations of the specimen surface after fatigue testing. The slip bands, and consequently the sites of cracks initiation, are mainly located in the austenite, and do not cover the ferrite in the low strain regime (Fig. 2(c)). This is consistent with the reported austenitic behaviour at low strain level. On the contrary, it is clear that both of the phases are involved in the accommodation of the plastic deformation at high strain amplitude (i.e. $\Delta e_t>1.3\%$, Fig. 2(d)). Moreover, the observations of short crack show that in this case the ferrite damages first what is in accordance with the reported ferritic behaviour at high strain amplitude.

#### 3.2. Fatigue Behaviour of the High Nitrogen Containing DSS

The general aspect of the stress responses to strain cycling of the DSS–0.4%N and of the DSS–0.24%N does not differ drastically from each other (Fig. 3(a)) i.e. hardening-softening behaviour depending on the strain amplitude. This feature is therefore generic for DSS. Meanwhile, a main difference concerns the stress values at the beginning of cycling which are higher in the DSS–0.4%N than in the DSS–0.24%N and which is related to the hardening influence of nitrogen. At last, the fatigue lives ($N_f$) of the DSS–0.4%N are slightly higher than those of the DSS–0.24%N.

A slope discontinuity, characterised by the Eqs. (3) and

### Table 1. Chemical composition of steels used for low cycle fatigue tests (wt%). (Fe bal.)

| Material     | N  | C  | Cr | Ni | Mn | Si | Mn | P | W  | Cr | S |
|--------------|----|----|----|----|----|----|----|---|----|----|---|
| DSS–0.24%N   | 0.24 | 0.018 | 24.81 | 6.56 | 3.71 | 0.36 | 0.98 | 0.023 | - | 1.62 | 0.0012 |
| DSS–0.4%N    | 0.40 | 0.023 | 25.2 | 7.1 | 4.1 | 0.24 | 2.90 | 0.015 | 0.57 | - | 0.001 |

![Fig. 1. Microstructure of the DSS–0.24%N alloy 50%α/50%γ (a) perpendicularly to the rolling direction (b) parallel to the rolling direction. Microstructure of the DSS–0.4%N alloy 30%α/70%γ (c) perpendicularly to the rolling direction (d) parallel to the rolling direction.](image-url)
Fig. 2. Results obtained on the DSS–0.24%N material: a) Cyclic accommodation curves; b) fatigue resistance curves; c) SEM micrograph of the surface of the failed sample tested at Δε=0.8% exhibiting slip bands essentially localised in the austenitic phase; d) SEM micrograph of the surface of the failed sample tested at Δε=2% exhibiting slip bands localised in both of the phases.

Fig. 3. Results obtained on the DSS–0.4%N material: a) Cyclic accommodation curves; b) fatigue resistance curves; c) SEM micrograph of the surface of the failed sample tested at Δε=0.8% exhibiting slip bands localised in the austenitic phase (60%) and in the ferritic phase (40%); d) SEM micrograph of the surface of the failed sample tested at Δε=2% exhibiting slip bands localised in both of the phases.
two phases steel is a bit more complex. Depending on the strain level, the actual behaviour of the interpretation based on a single well defined phase response, 0.91 for Eqs. (1) and (2).

With a regression coefficient of 0.953 instead of 0.997 and 0.889 for Eqs. (3) and (4).

Indeed, the microscopic observations of the plasticity clearly show that, at low strain amplitude, the plastic deformation is accommodated by both of the phases in the DSS–0.4%N, even if the majority of the slip bands are displayed in the austenite. Moreover, as can be seen in Fig. 3(c), it is now possible to observe some cracks in the ferrite what was not the case in the DSS–0.24%N. At high strain amplitude both of the phases accommodate the plastic deformation and the ferrite still cracks at first as for the DSS–0.24%N (Fig. 3(d)).

As a consequence, the slope discontinuity cannot be any longer simply associated with the existence of two distinct behaviours (ferritic at high $\Delta e$, austenitic at low $\Delta e$) and this explanation is not sufficient and above all inexact in the case of high nitrogen duplex stainless steels. This suggests that the description of the fatigue resistance of these DSS with two equations does not necessarily reflect an unambiguous physical meaning but it allows a better prediction of the fatigue life.

For the DSS–0.4N, a single fitting lead to the following equation:

$$
\Delta e_p = 32 \cdot N_t^{-0.5} \quad \text{(DSS–0.4%N)} \quad \text{......(5)}
$$

with a regression coefficient of 0.902 instead of 0.997 and 0.889 for Eqs. (3) and (4).

Similarly, for the DSS–0.24N, we obtain:

$$
\Delta e_p = 39 \cdot N_t^{-0.51} \quad \text{(DSS–0.24%N)} \quad \text{......(6)}
$$

with a regression coefficient of 0.953 instead of 0.96 and 0.91 for Eqs. (1) and (2).

These results show that despite the consistency of the interpretation based on a single well defined phase response, depending on the strain level, the actual behaviour of the two phases steel is a bit more complex.

3.3. Influence of the Environment

In order to evaluate the sensitivity of each phase against deformation and environment, corrosion fatigue tests have been carried out on both of the materials. They have been performed at free potential, at room temperature, in an aqueous solution containing 30 g of NaCl per litre of water. Since the specimen was immersed in the solution, deformation has been measured by an external rigid mechanical device.

As expected, the stress response $\sigma(N)$ to strain cycling in the NaCl containing solution does not differ significantly from that in air tests (Fig. 4(a)), meanwhile the environment affects the specimen surface but does not modify the macroscopic response. However, since cracking occurs at surface, the fatigue resistance may be modified. The results show that the DSS–0.24%N alloy is very sensitive to the corrosive media since a systematic reduction (about twice lower) of the fatigue lives can be noticed (Fig. 4(b)). In contrast with the DSS–0.24%N steel, the DSS–0.4%N alloy is differently affected by the corrosive environment. Indeed, the reduction in fatigue life progressively occurs with increasing the strain range. No effect is observed for the lowest strain test. In this respect, for the DSS–0.24%N alloy, the exponent value of the Coffin Manson relationship for the corrosive environment tests (Eq. (7)) is close to the average value obtained for air tests (Eq. (5)).

$$
\Delta e_p = 38 \cdot N_t^{-0.56} \quad \text{(DSS–0.24%N)} \quad \text{......(7)}
$$

This is not the case for the DSS–0.4%N alloy (Eq. (8) to be compared with Eq (6)):

$$
\Delta e_p = 10 \cdot N_t^{-0.37} \quad \text{(DSS–0.4%N)} \quad \text{......(8)}
$$

The detrimental effect of the corrosive environment which appears at high strain level disappears at low strain amplitude. Therefore, as shown in Fig. 4, a threshold of strain amplitude (near $\Delta e_p<0.3\%$ corresponding to $\Delta e<1\%$) appears where there is some interest to use the DSS–0.4%N alloy.

Metallographic observations of the specimen surfaces observed after failure show that both of the phases contain fatigue extrusions and cracks in the ferritic grains. The surface topography was more marked for the tests in corrosive environment than in air (Fig. 5).
4. Discussion

4.1. Role of Nitrogen

On sight of the results, nitrogen addition in duplex stainless steels affects the fatigue properties but the changes cannot be simply deduced from a parallel with those induced by nitrogen alloying of single phase austenitic steel. First of all, because of the very different solubility of nitrogen in ferrite and austenite, the partition coefficient, ratio of nitrogen concentrations in α and γ, is very far from unity and therefore the nominal N concentration is in DSS nearly half of that of austenite and depends on the austenite/ferrite volume fraction. For steels DSS–0.24%N and DSS–0.4%N studied here, the nitrogen content in the austenite is 0.44wt% for the DSS–0.24%N while 0.54 wt% for the DSS–0.4%N. In this way, alloying with nitrogen a DSS will emphasise the behaviour of austenite, as shown in the present study by the yield stress and the cyclic stress values measured at the very first cycles of the tests which are always higher in the case of the DSS–0.4%N containing the largest amount of nitrogen (Table 2). This reflects the strong solution hardening effect produced by nitrogen. Nevertheless, after a given number of cycles, the “stabilised” value of the stress amplitude (noted σa), sometimes conventionally defined for Nf/2 and also labelled “cyclic stress”, is similar for both of the steels because of the softening. This softening also reflects the effect of nitrogen which promotes planar slip of dislocations in the austenite6) which in turn favours the reversibility of the plastic cyclic deformation and therefore the softening of the austenite and then of the steel. Nevertheless, it should be mentioned that the here studied steels do not only differ in their chemical composition but also in their rolling process. In order to take into account this effect due to the rolling process, additional investigations have been carried out on another DSS alloyed with 0.25% nitrogen and elaborated as the DSS–0.4%N steel (more details are reported elsewhere7,8). Figure 6 contains the values of the cyclic stress amplitude measured at mid-life as a function of the nitrogen content for these two materials that have undergone the same elaborating process. The hardening effect of nitrogen is more evidenced. Indeed, the cyclic stress values (σa) measured on the 0.4% nitrogen containing DSS at each strain level are systematically higher than those observed on the 0.25% N DSS. It is also noticeable that the increase of the austenite fraction up to 70%, corresponding to the γ-stabiliser effect of the nitrogen, leads to a decrease of the softening rate after cycling from theoretically 17.5 to 13% (Data at 0.15% N corresponds to Refs. 4) and 11)).

![Figure 5](image_url)

**Fig. 5.** Corrosion fatigue results. Typical topography obtained after fatigue on a failed sample of (a) DSS–0.24%N tested at Δεf=1.6% and (b) DSS–0.4%N tested at Δεf=1.2%.

| Δεf (\%) | σy (MPa) | σmax (MPa) | σa (MPa) |
|----------|-----------|-------------|-----------|
| DSS–0.24%N | DSS–0.4%N | DSS–0.24%N | DSS–0.4%N | DSS–0.24%N | DSS–0.4%N |
| 2        | 659       | 705         | 695       | 740       | 631       | 630       |
| 1.6      | 647       | 683         | 690       | 725       | 617       | 617       |
| 1        | 591       | 631         | 610       | 660       | 550       | 565       |
| 0.8      | 560       | 573         | 592       | 614       | 535       | 532       |

σ1/4 is the stress measured after the first quarter of cycle, σmax is the maximal stress amplitude measured at the beginning of cycling and σa is the stress amplitude measured at mid-life.

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![Figure 6](image_url)

**Fig. 6.** Effect of nitrogen on the material hardening: the cyclic stress values measured on the 0.4% nitrogen containing DSS at each strain level are systematically higher than those observed on the 0.25% N DSS. It is also noticeable that the increase of the austenite fraction up to 70%, corresponding to the γ-stabiliser effect of the nitrogen, leads to a decrease of the softening rate after cycling from theoretically 17.5 to 13%. (Data at 0.15% N corresponds to Refs. 4) and 11)).
investigation should be carried out with the procedure employed for single phase austenitic stainless steels. Moreover, the increase in fatigue resistance must not be evaluated only from the Coffin–Manson relationship but should also take into account the stress levels. In the present study, both of the DSS–0.24%N and DSS–0.4%N alloys exhibit at the same strain level nearly the same fatigue lives but cyclic stress amplitude is much higher in the DSS–0.4%N which makes this steel attractive for application.

The last situation where nitrogen can influence the fatigue resistance is in presence of a corrosive environment. First, it must be recalled that ferrite being less noble than austenite, the latter is cathodically protected by the former in the undeformed state. In chloride media, nitrogen improves corrosion resistance of austenitic stainless steels especially pitting corrosion when alloyed with molybdenum. That is increasing nitrogen content in a DSS magnifies the protection of the austenitic phase by the ferrite. This can account for the difference in fatigue lives in the considered strain range. In this range where most of the slip bands occur in the austenite, anodic sites can appear but a rapid repassivation is easier in the high nitrogen containing austenite of the DSS–0.4%N which avoids a loss in corrosion-fatigue performance.

4.2. Effect of the Phase Volume Fraction

In the present study, the difference in phase volume fraction is correlated with the strong effect of nitrogen to promote the austenitic phase, therefore, the effect of phase volume fraction on fatigue properties is accompanied by the effect of the N on the fatigue behaviour of each α and γ phase. A previous study on the low cycle fatigue of a DSS containing 0.15% N has pointed out that the macroscopic mechanical response as well as the dislocation microstructures inside each phase is not simply the consequence of an individual evolution of the microstructure in each phase independently, but also results from a mutual interaction between austenite and ferrite. This refers to a coupling effect which more intensively occurs at high strain amplitude. It has been suggested that a load transfer from the austenite to the ferrite progressively occurred during cycling as result of the austenite which cyclically softened. The softening effect related to the occurrence of planar dislocation arrangements in the austenite is to some extent controlled by the nitrogen content in the austenitic phase, it is also due in DSS to the volume fraction of austenite. As can be seen in Fig. 6, the softening rate δ of the DSS–0.4%N is 13% with a mixture of 70%γ/30%α instead of an estimated value of 17.5% if the alloy had a mixture of 50%γ/50%α. It is reasonable to suppose that, if the softening is less pronounced in the austenite then the load transfer is reduced. Though some slip bands can be observed at low strain amplitude in the ferrite, this avoids a progressive input of strain in this phase which would earlier crack and allows to keep a good fatigue resistance of the DSS–0.4%N.

The effect of volume fraction is emphasised when cycling in the NaCl water solution. Nucleation growth of short cracks in the ferrite is facilitated by the dissolution of the matrix and the weak possibility to repassivate the anodic sites. The reduction in fatigue resistance of the DSS–0.24%N results from the easy coalescence of these short cracks. The coalescence can be delayed if the plastic zones at the crack tip do not overlap. The mean distance between two cracks coming from the ferrite and likely to coalesce in the austenite is larger with increasing the volume fraction of austenite which confers to the DSS–0.4%N a better corrosion-fatigue resistance at low strain amplitude than to the DSS–0.24%N. It has been shown that similar arguments can be used for obtaining DSS with a fatigue resistance weakly, or even not, sensitive to “475°C embrittlement” heat treatments.

5. Conclusions

The low cycle fatigue behaviour of two DSS alloyed with 0.24% N and 0.4% N has been studied in air and in a NaCl water solution. The main conclusions are:

(1) The cyclic response of high nitrogen (0.25–0.4%) duplex stainless steels does not qualitatively differ from that of lower nitrogen content (0.072–0.15%) and consists of a hardening followed by a softening stage before stabilising.

(2) Nitrogen addition leads to monotonic hardening but to cyclic softening of the austenite.

(3) The effect of nitrogen cannot be evaluated from the nominal composition of the steel but from the local one in the austenitic phase.

(4) The DSS 0.4% N alloyed exhibits a higher stress amplitude and better fatigue resistance especially in a NaCl water solution than the DSS alloyed with 0.24% N and therefore is interesting for application.

(5) In a NaCl water solution, the ferrite is the phase in which initiation and growth of short cracks is promoted by the environment.

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