



# Cyclic behavior of a superferritic stainless steel at room and intermediate temperatures



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## ABSTRACT

This paper studies the fatigue behavior at room and intermediate temperatures of a superferritic stainless steel UNS S 44600 that had received different heat treatments. Thermal treatments at high temperature followed by water quenching produces needle-like Cr<sub>2</sub>N precipitates that detrimentally affect the fatigue life of superferritic stainless steels. At intermediate temperatures, independent of previous thermal treatments, the occurrence of dynamic strain aging was manifested by a pronounced cyclic hardening rate, inverse dependence of the peak tensile stress with strain rate, a high dislocation density and modified dislocation arrangements. Cycling at intermediate temperature demodulates the spinodal decomposition with a fast velocity, improving the fatigue life of aged samples.

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## 1. Introduction

Superferritic stainless steels contain high levels of Cr (22–3 wt.%), Ni and Mo (1–5 wt.%) and small quantities of interstitial elements (C and N <0.05 wt.%). In recent years, the worldwide demand has driven nickel and molybdenum prices to record high values. These steels, with low Ni content and reasonable Mo content, are a cost effective alternative that have attractive mechanical properties, high thermal conductivity and good corrosion resistance. Thus, these alloys are finding application in heat exchangers, the petrochemical industry and desalination and water recovery [1]. However, the ductility of these steels could be seriously weakened in intermediate temperature applications. Thus, the low cycle fatigue failure prevention is an important issue for structural components of these steels used at intermediate temperatures. In this sense, the phenomenon of dynamic strain aging (DSA) in ferritic stainless steels has been investigated in tensile and fatigue tests within the 300–600 °C range [2–4]. DSA occurs when solute atoms possess enough mobility to catch up with the moving dislocations and reduce dislocation motion during deformation. As DSA develops when the diffusivity of the solute atoms approaches the dislocation velocity, its occurrence depends on temperature and strain rate. During cyclic tests the usual manifestations of DSA are: an unusual increase in cyclic hardening rate, inverse dependence of the peak tensile stress with strain rate, plastic-deformation insta-

bility, enhanced dislocation density and modified dislocation arrangements [2–8]. On the other hand, in ferritic stainless steels aging between 400 °C and 500 °C leads to the well known “475 °C embrittlement” [9,11]. The “475 °C embrittlement” hardens and embrittles the steels at room temperature and it is believed to be caused by the spinodal decomposition (SD) of the ferrite phase into Cr-rich and Fe-rich phases [11].

Although for ferritic stainless steel all the possible embrittlement mechanisms are well-identified in literature, many doubts still remain on their consequences for cyclic deformation and fracture. The present paper aims to study the fatigue behavior of a superferritic stainless steel at room and intermediate temperatures after different thermal treatments. In doing so, at intermediate temperatures, the existence of a dynamic strain aging phenomenon (DSA) and the effect of fatigue on SD are evaluated.

## 2. Material and experimental procedure

The material used in this investigation is a UNS S 44600 superferritic stainless steel with the chemical composition given in Table 1. The steel was received in the form of hot-rolled cylindrical bars of 25.4 mm diameter. During the fabrication processes of the bars it is possible that an inhomogeneous dislocation structure and harmful phases such as chromium carbides or  $\sigma$  phase were formed [12]. Reheating ferritic stainless steels between 950 and 1100 °C and then cooling rapidly will dissolve the chromium carbides and  $\sigma$  phase and prevent re-precipitation during cooling [13,14]. However, these thermal treatments can also form

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**Table 1**  
Chemical composition of UNS S 44600 (wt.%).

C	Cr	Mn	Cu	Si	V	Sn	N	S	Ni	P	Nb	Mo
0.022	23.44	0.75	0.08	0.29	0.12	0.008	0.062	0.002	0.3	0.019	0.1	0.08

**Table 2**  
Thermal treatments applied to UNS S 44600.

TTW	1100 °C for 30 min followed by water quenching
TTA	1100 °C for 30 min followed by air cooling
TTW + AG	1100 °C for 30 min followed by water quenching and then heat treated 100 h at 475 °C
TTA + AG	1100 °C for 30 min followed by air cooling and then heat treated 100 h at 475 °C

chromium nitrides, if free nitrogen is present in the steel matrix. These precipitates detrimentally affect the mechanical properties and decrease fracture toughness at low temperature [15]. Therefore, two homogenization thermal treatments at 1100 °C for 30 min were carried out in the as-received bars, one ended with water quenching (TTW) and the other with air cooling (TTA). After the TTW the grain size was  $(77 \pm 6) \mu\text{m}$  whereas after TTA was  $(81 \pm 5) \mu\text{m}$ . An additional aging of 100 h at 475 °C were given to some TTW and TTA bars. This material is referred as aged (AG). Table 2 shows the different thermal treatments. Push-pull cylindrical specimens with a gauge length of 18.4 mm and a diameter of 5 mm were machined from the thermally treated bars.

Cyclic deformation tests were carried out with an Instron (model number 1362) electromechanical testing machine under plastic strain control using a fully reversed triangular form signal. The specimens were tested in air between 20 and 475 °C with a plastic strain range of 0.3% performed at a total strain rate of  $2 \times 10^{-3} \text{ s}^{-1}$ . Between 20 and 500 °C, the strain rate dependence of the peak tensile stress was evaluated by changing the strain rate one order of magnitude during cyclic tests at a total strain range of  $\Delta\epsilon_t = 1.14\%$ . The experimental procedure, already used in other materials [5–7], consisted of: at each temperature, starting at 20 °C, the sample was allowed to reach approximately 300 cycles with a strain rate of  $2 \times 10^{-3} \text{ s}^{-1}$ . This number of cycles corresponds to an almost stabilized peak tensile stress ( $\sigma_1$ ) in most of the tests performed at different temperatures. Then, without any interruption of the test, the strain rate was decreased to  $2 \times 10^{-4} \text{ s}^{-1}$  and after reaching stabilization, a peak tensile stress ( $\sigma_2$ ) was measured. Stress increments were measured in the form  $\Delta\sigma_{\text{strain rate}} = \sigma_2 - \sigma_1$ . This procedure was repeated with increasing temperatures.

The microstructure and precipitates were examined using optical microscopy. Transmission electron microscopy (TEM) equipped with energy dispersive spectroscopy (EDS) was used to make a detailed study of the size and composition of the precipitates. Both replica and thin foil transmission electron microscopy techniques were used to identify the precipitates.

After fatigue tests, the fracture surfaces were analyzed using SEM. Dislocations structures were observed in a transmission electron microscope (TEM).

### 3. Results and discussion

#### 3.1. Thermal treatments

The homogenization thermal treatments applied in this work are reported to be sufficient for preventing the formation of harmful  $\text{M}_{23}\text{C}_6$  and sigma phase in ferritic stainless steels containing V and Nb [14]. Optical micrographs of the TTW samples show

numerous small precipitates, with sizes of approximately 0.5  $\mu\text{m}$ , distributed mainly inside the ferrite grains, Fig. 1a. New precipitates, too thin to be detected by optical microscopy, with a needle-like morphology are observed by TEM in the TTW samples, Fig. 1b. In Fig. 1c these needle-like precipitates are clearly visible in the dark field image (DF). This DF image was obtained using a lattice reflection of a hexagonal closed-packed structure, which is contained within the BCC ferrite phase, Fig. 1d. An EDS's analysis of extracted precipitates revealed the existence of two types of precipitates. In this sense, in TTW samples in addition to the expected NbC precipitates [14], Fig. 1e, other thin precipitates with high contents of Cr and N were detected, Fig. 1f. In ferritic stainless steel as well as in the ferrite phase of duplex stainless steel [15–17], particles identified as  $\text{Cr}_2\text{N}$  are formed after quenching from higher temperatures. Usually, precipitates of this kind occur at aging temperatures between 700 and 900 °C, but they have been found as a result of fast cooling from higher temperatures. Higher solution temperatures increase the solubility of nitrogen in ferrite and higher cooling rates improve the driving force for hexagonal closed-packed  $\text{Cr}_2\text{N}$  formation as a result of the supersaturation of nitrogen in the ferrite. According to Safsten [16], depending on the cooling rate different morphologies of  $\text{Cr}_2\text{N}$  can be observed. As the cooling rate increases, the precipitates' morphology changes from needle to cloud like. Hence, from the observed diffraction pattern, Fig. 1d, and the EDS measurements, Fig. 1f, it can be confirmed that after TTW needle-like  $\text{Cr}_2\text{N}$  precipitates are present in this super-ferritic stainless steel. Demo [18] reported that in AISI 446 ferritic stainless steel the precipitation of nitrides at grain boundary did not strongly affect the toughness, whereas, fine nitride precipitation on dislocations degrades the ductility of this steel. These precipitates reduce the dislocation mobility, decreasing the toughness of the materials. Nevertheless, no information about how  $\text{Cr}_2\text{N}$  precipitates affects fatigue has been reported for superferritic stainless steel. Attempting to avoid  $\text{Cr}_2\text{N}$  precipitation, TTA was employed. Fig. 2a shows the macroscopic structure of specimens with TTA. It turned out that the size of precipitates has increased significantly in comparison with TTW. Additionally, TEM images of TTA samples, Fig. 2b, give no evidence of needle-like precipitates. EDS's results of TTA samples show the presence of NbC as in TTW, but with a greater size (approx. 1.5  $\mu\text{m}$ ). Moreover, VC precipitates are also found, Fig. 2c.

In both TTW and TTA aged samples, a mottled contrast, typical of the presence of the spinodal decomposition, is clearly visible, Fig. 3.

#### 3.2. Fatigue behavior

##### 3.2.1. Cyclic response at 20 °C

Fig. 4 shows the cyclic response of the superferritic stainless steel UNS S 44600 at 20 °C after different thermal treatments. The TTW material attains a higher stress level than the TTA. On the other hand, as a consequence of aging, the highest stress levels were observed in the AG samples. The cyclic behavior of this steel, with TTA and TTW, is characterized by an initial cyclic hardening followed by cyclic softening, which ends in a saturation stage. In contrast, both the hardening and softening of AG samples are more pronounced and the saturation stage is never reached. Instead, an abrupt failure of the specimen is observed. The strong embrittlement of ferritic stainless steel in fatigue testing due to the SD is

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