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Secondary Hardening Behavior in Super Duplex Stainless Steels during LCF in Dynamic Strain Ageing Regime

Guocai Chai[a,b*], Marcus Andersson[a]

[a]Sandvik Materials Technology, R&D center, 811 81 Sandviken, Sweden
[b]Division of Engineering Materials, Linköping University, SE-58183 Linköping, Sweden

Abstract

Cyclic deformation behaviors in five modified duplex stainless steel S32705 grades have been studied at 20°C, 200°C, 250° and 350°C. The influence of temperature and nitrogen concentration on the occurrence of the second hardening phenomenon, in the stress response curve was focused. An increase in nitrogen concentration can have a positive effect on dynamic strain ageing by increasing the first hardening and also the second hardening behavior during cyclic deformation. Furthermore, an increase in nitrogen concentration in the super duplex stainless steel increases the fatigue life in the strain ageing temperature range. The occurrence of strain ageing in duplex stainless steel has greatly changed dislocation structures. The formation of irreversible dislocation structures and stacking faults can contribute to the formation of second hardening in the stress response curve.

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Keywords: Duplex stainless steels; static and dynamic strain ageing; nitrogen content; mechanical behavior; ductility

1. Introduction

As known, the diffusion of carbon and nitrogen atoms in metals in the temperature range 150 – 450ºC may lead to strain ageing. It is a phenomenon that these interstitial atoms interact with moving dislocations in steels or other alloys with small amounts of interstitial elements [1, 2]. Strain ageing is usually divided into two types: static strain aging (SSA) and dynamic strain aging (DSA). Static strain aging is a hardening phenomenon that occurs in a cold plastic deformed material during ageing. This will consequently cause an increase in the yield strength. Dynamic strain ageing is the interaction between dislocation movement and interstitial atoms in steels. They occur during the deformation process depending on temperature and strain rate. A typical characteristic is the formation of serrated stress versus strain curves, which has been called the jerky flow or the Portevin-LeChatelier (PLC) effect [3]. DSA can also lead to an increase in flow stress, work hardening rate and more importantly, and a negative strain rate sensitivity. Strain ageing is believed to cause an embrittlement in metals [1], and becomes a concern for material applications.

* Corresponding author:
E-mail address: guocai.chai@sandvik.com

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There has been extensive work on DSA over the last few decades [1]. Recently, DSA in duplex stainless steels has been investigated by tensile tests [4] and low cycle fatigue tests [5] at temperatures ranging from 150 to 500°C. The maximum DSA effect was observed at 325 – 350°C [6]. A secondary hardening was observed in the temperature range from 200 – 440°C [5]. However, the mechanisms for the second hardening have not been explained. This paper provides an investigation on the influence of nitrogen content on the dynamic strain ageing behavior in five modified super duplex stainless steel S32705 grades at 350°C. The mechanisms for the second hardening will be discussed.

2. Materials and experimental

Five modified duplex stainless steel S32705 grades were prepared. These alloys have three nitrogen content levels and three ferrite contents. The detailed information is shown in Table 1.

Table 1. Modified duplex stainless steel S32705 grades (wt.%).

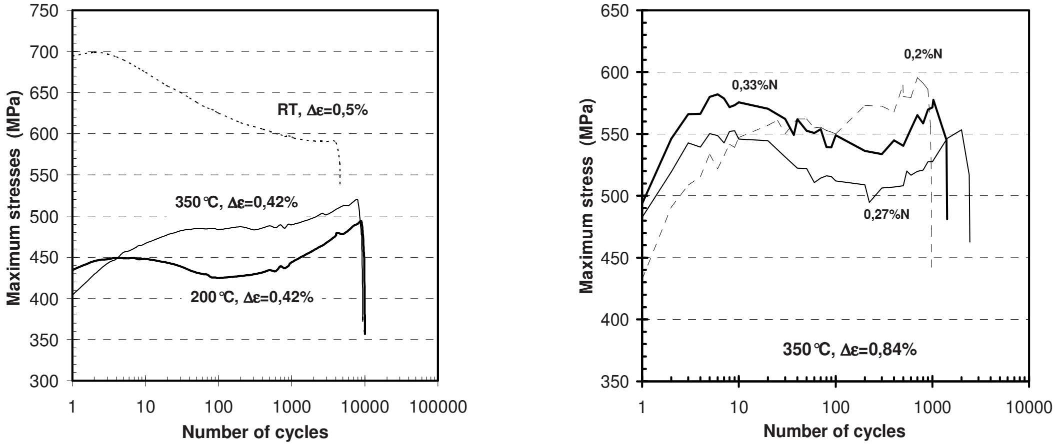
Grades	Dimension	C	Si	Mn	Cr	Ni	Mo	N	PRE	Ferrite	Rp0,2	Rm	A
										(%)	(MPa)	(MPa)	(%)
25-A	d=20mm	0.01	0.34	0.33	24.9	6.98	3.69	0.20	40	49	453	710	38.8
25-B	d=20mm	0.01	0.26	0.29	24.8	6.83	3.75	0.33	42	37	464	764	42.4
25-C	d=20mm	0.01	0.33	0.29	25.0	8.19	3.67	0.21	40	37	455	726	39.8
25-D	d=20mm	0.01	0.26	0.28	25.0	5.54	3.74	0.33	43	49	492	765	40.3
25-E	d=20mm	0.01	0.30	0.30	25.0	6.91	3.92	0.27	42	44	461	746	40.5

For dynamic strain ageing test, cylinder-shaped samples with a diameter of 10 mm and a length of 12.5 mm were prepared from modified S32705 bar-shaped grades with a diameter of 25 mm. The dynamic strain ageing tests were carried out in a computer-assisted Instron 1342 servo-hydraulic machine. The tests were performed at RT (20°C), 200°C, 250°C and 350°C using a heating resistance chamber with a temperature accuracy of 1°C. The total strain amplitude ranged from 0.4 to 1.0% with a push-pull mode and a frequency of 0.15Hz.

The dislocation structures were investigated using transmission electron microscopy (TEM). Discs with a diameter of 3 mm were taken from both the fatigue-tested specimens and as-received material for TEM investigation. Thin foils were electrochemically polished at -30°C using an electrolyte of 10% perchloride acid ($HClO_3$) in methanol and a voltage of 17-18 V. In order to reduce the risk for surface absorption of carbon, the fresh thin foils were immediately inserted into sample holder and analysed. The dislocation structures were studied using a Philips CM200 FEG-TEM, operated at 200 kV.

3. Results and discussion

Figure 1 shows the stress response curves for grade 25-E. As expected, the cyclic stress response of this duplex grade shows an initial hardening followed by a continuous softening behavior at RT (Fig. 1a). At 350°C, however, it shows a very different behavior: a continuous hardening behavior until damage or failure. At 200°C, a second hardening occurs after about 100 cycles until damage or failure has occurred. The stress response curve at 350°C shows a higher cyclic hardening rate in the first hardening period compared with the curves at RT and 200°C. This indirectly shows the effect of dynamic strain ageing. The occurrence of cyclic strain hardening and softening strongly depends on the dislocation multiplication, accumulation rate and dislocation annihilation rate in the metals during cyclic loading. This indicates that the cyclic strain hardening in the initial cycles is due to the dislocation multiplication and accumulation within the initiated slip bands. With further fatigue cycling, the annihilation rate increases due to an increasing dislocation density. This can lead to a progressive decrease in the strain hardening rate and finally a state of saturation, which results in softening as the dislocation annihilation rate is higher than the dislocation accumulation rate. An increase in temperature will increase dislocation annihilation, and consequently lead to an earlier softening and a higher softening rate. However, an increase in temperature can greatly increase diffusion rates of carbon and nitrogen atoms, which increases the interaction between dislocation movement and the interstitial atoms or the occurrence of dynamic strain ageing. Jagged curves have been observed in this study (Fig. 2). This can greatly reduce the dislocation annihilation rate and consequently increase the cyclic hardening rate. When the dynamic strain ageing is so effective that dislocation annihilation rate can be greatly reduced, only cyclic hardening can occur.



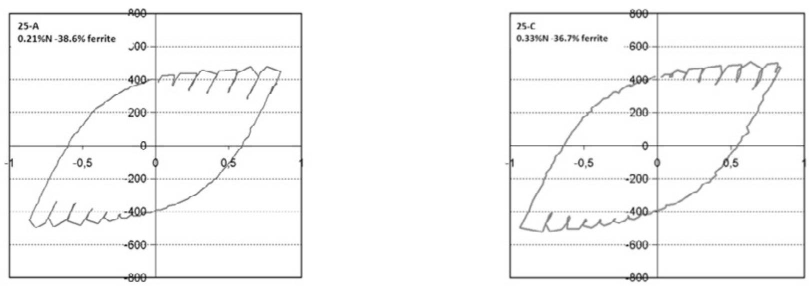
(a) (b)

Fig. 1. Stress response curves in modified super duplex stainless steel S32705 grades, (a). Influence of temperature; (b). Influence of nitrogen contents.

Table 2 shows the influence of temperature (25-350°C) on the fatigue life of grade 25-E during cyclic loading with different strain amplitude. No detrimental effect of dynamic strain ageing can be observed. It is just the opposite. The fatigue life of the material in the dynamic strain ageing temperature range is higher than that at 25°C. One reason is that dynamic strain ageing in DSS can increase both strength and elongation [8].

Table 2. Influence of strain and temperature on fatigue life (number of cycles).

Temperature (°C)	Strain amplitude (%)			
	0.4	0.42	0.7	0.84
RT	7867		2333	
200		10032		1697
250	11638		2665	
350		9394		2456



(a) (b)

Fig. 2. Response jagged curves from the 2nd cycles for Alloy 25-A and Alloy 25-C during cyclic loading at 350°C.

Fig. 1b shows the influence of nitrogen content on the cyclic stress response for grades 25-A, 25-D and 25-E with a strain amplitude of 0.84% at 350 °C. The specimen with the highest nitrogen content (25-A) shows higher first and second hardening responses compared with specimen 25-D, and even longer fatigue life (with similar ferrite content). Grade 25-E has a nitrogen content of 0.27% and a ferrite content of 44%, which is a lower ferrite content than grade 25-A and 25-D. This alloy shows the longest fatigue life, which indicates that both nitrogen and ferrite contents are important for the fatigue life. The nitrogen content has strongly affected the stress response behavior in this duplex stainless steel during dynamic strain ageing. The alloy with low nitrogen content shows almost a continuous hardening behavior during the whole process. For the alloys with higher amount of nitrogen, a softening process can be observed. The main reason for this could be that the nitrogen content in the alloys strongly affects the first cyclic hardening rate. The hardening rate increases with increasing nitrogen content in the alloys by increasing the dislocation pinning effect, which consequently greatly increases dislocation density in the alloys. However, a high dislocation density increases recovery or dislocation annihilation rate during cyclic loading. This indicates that an increase in nitrogen content in duplex stainless steels can cause cyclic softening if the first hardening rate is rather high.

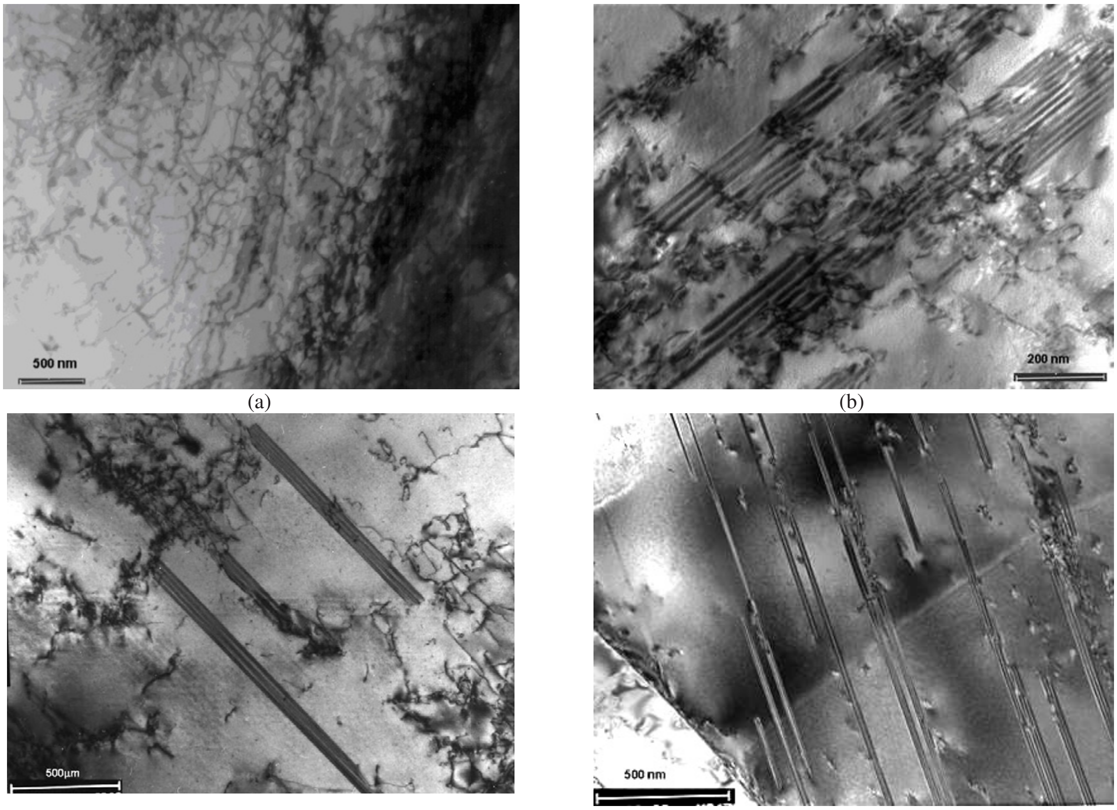


Fig. 3. Dislocation structures, (a). Planar dislocation in the austenitic phase, $\Delta\varepsilon/2$=0,6%, 4608 cycles, at RT; (b). Interaction between dislocations and stacking faults in the austenitic phase, $\Delta\varepsilon/2$=0,84%, 2456 cycles, at 350°C; (c). 0,21%N, austenite, $\Delta\varepsilon/2$=0,84%, 1486 cycles, , 350°C; (d). 0,33%N, austenite, $\Delta\varepsilon/2$=0,84%, 1417 cycles, 350°C..

In order to investigate some possible mechanisms for the second cyclic hardening in the stress strain response curve, the dislocation structures in the tested specimens have been studied. In the specimen tested at RT, dense tangled dislocations in the ferritic phase and planar dislocations with low density in the austenitic phase (Fig. 3a) are typical structure. In the specimens tested at 350°C, dislocation sub-cells with high dislocation density can be observed in the ferritic phase, and planar dislocations and stacking faults with high density can be observed in the austenitic phase. Fig. 3b shows one example of interactions between the moving dislocations and stacking faults. Nitrogen content in the alloy has a strong effect on the dislocation structures. For the DSS with low nitrogen content, the planar dislocation structure has few and not well organized dislocations. Dislocation bands with high density can be observed in the austenitic phase. In the ferritic phase, tangled dislocations are a typical structure. In these alloys, few stacking faults have also been observed (Fig. 3c)). This planar dislocation structure becomes denser and well organized if the DSS contains higher nitrogen content or is tested at 350°C. This indicates that dynamic strain ageing with high nitrogen will promote the formation of planar dislocation structure, which leads to a cyclic hardening [6]. Now more fine stacking faults or probably twins can be observed (Fig. 3d). They are usually interacted with moving dislocation. In the ferritic phase, dislocation sub-cells with high dislocation density are more easily observed. From this dislocation structure analysis, the formation of the second hardening can still be attributed to the formation of irreversible dislocation structures and defects like stacking faults. The first cyclic hardening rate is also a critical parameter that affects the softening process.

This study has showed that DSA in DSS may change the dislocation structure, which leads to the formation of stacking faults or twins. This indicates that the stacking faults energy can decrease in the temperature range of 25 - 350°C. Stacking faults usually appear in FCC metals with low stacking fault energy during plastic deformation and an increase in nitrogen content decreases the stacking fault energy in austenitic steels [7].

For DSS, spinodal decomposition can occur at temperature near 470°C. At 350°C, the occurrence of the spinodal decomposition can be expected very slow, which may not affect the DSA significantly.

4. Conclusions

Dynamic strain ageing in duplex stainless steels during cyclic loading causes the formation of a second hardening response in the temperature range of 150 - 350°C and the fatigue properties improves compared to RT.

An increase in nitrogen content in duplex stainless steel can increase the dynamic strain ageing effect and fatigue life in the strain ageing temperature range. The occurrence of strain ageing can change dislocation structures. The formation of irreversible dislocation structures and stacking faults can contribute to the formation of the second hardening in the stress response curve.

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