Change of stress-strain hysteresis loop and its links with microstructural evolution in AISI 316L during cyclic loading

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Abstract

The cyclic deformation behaviour of an austenitic stainless steel and its links with microstructure are examined by analysing changes in the stress-strain hysteresis loop characteristics and dislocation condition with increasing cycle number. In terms of peak tensile stress, AISI 316L exhibits a hardening stage followed by a softening stage, and finally a stable response stage, during strain-controlled cyclic loading. During the hardening stage, dislocation density increases, mainly in the form of planar structures, i.e. stacking faults, pile-ups. Dislocations then rearrange to form walls and channels during the softening stage, which further develop to become a cellular structure later in this stage and during the final stable response stage. An analysis of the change in hysteresis loop shape during cyclic loading shows that the increase in dislocation density is responsible for an increase in effective stress. In addition, the long range internal stress (back stress), which is strongly sensitive to dislocation distribution and dislocation structure, is mainly responsible for the cyclic deformation response. The loop shape parameter and other quantities derived from the stress-strain hysteresis loop also evolve along with the development of the dislocation condition.

Keywords: AISI 316L; Low cycle fatigue; Hysteresis loop; Dislocation evolution.

1. Introduction

Austenitic stainless steels are widely used in industrial applications due to their outstanding corrosion resistance at low temperatures, and high oxidation resistance and superior creep-fatigue properties at high temperatures. It is important to understand the material characteristics responsible for their performance...
in service, in particular their cyclic deformation response. Parameters representing cyclic mechanical behaviour and its relationship to microstructural condition in strain-controlled low cycle fatigue (LCF) tests can be obtained by determining characteristic features of stress-strain hysteresis loops such as the peak stresses, the effective stress, the back stress and the loop shape parameter [1-6]. Diagrams of peak stress versus number of cycles indicate if a material exhibits either a cyclic hardening or cyclic softening stress response, or both, before a possible saturation stage. Effective stress and back stress (i.e. the internal stresses) which associate with the short-range and long-range order of dislocation interactions provide information concerning the relationships between mechanical behaviour and microstructural condition during cyclic loading [1, 3, 7, 8]. Moreover, loop shape parameter, can indicate how the microstructural condition of the material evolves during loading [5].

While the analysis of stress-strain hysteresis loops gives an understanding of how materials behave during cyclic deformation at a macroscopic scale, microstructural characterisation provides an insight understanding of the evolving microstructures during cyclic loading. By appropriate correlations, relationships between mechanical properties and microstructural condition may be revealed. There have been intensive studies of the relationship between cyclic deformation properties and the dislocation structures existing at the saturation stage or at failure of austenitic stainless steels over a range of strain amplitudes [9-18]. However, the fatigue response of austenitic stainless steels, like other metals [19], significantly changes during the first cycles. Consequently, the relationship between the cyclic deformation response of austenitic stainless steels and microstructural condition changes also considerably during early cyclic loading, in a way which can strongly affect the later fatigue response of this class of materials. As a result, the investigation of changes in cyclic deformation response and microstructural condition during the course of loading is essential.

In this study, a test condition responsible for an austenitic stainless steel (AISI 316L) exhibiting cyclic hardening, cyclic softening and a stabilised stress response has been studied. Cyclic deformation response of the material is investigated by analysing changes in the stress-strain hysteresis loop through three stress response stages. The evolution of microstructure during cyclic loading has been carefully studied in order to establish the relationship between the cyclic deformation behaviour and microstructural condition.

2. Experimental procedure

The composition of the AISI 316L steel evaluated in this study is shown in Table 1. The material originated from a hot finished primary cooling circuit pipe which had been quenched in water from 1050/1080°C. This solution treatment was responsible for an austenite grain size of 60 μm.

| C (%) | Mn (%) | P (%) | S (%) | Cr (%) | Mo (%) | Ni (%) | Cu (%) | B (%) | Co (%) | N (%) |
|-------|--------|-------|-------|--------|--------|--------|--------|-------|--------|-------|
| 0.014 | 1.630  | 0.030 | 0.002 | 16.730 | 2.120  | 11.090 | 0.600  | 0.0011 | 0.090  | 0.060 |

Cylindrical testpieces, with a parallel length of 20mm, a gauge diameter of 8mm and with threaded end-grips, were machined with an axial orientation from the pipe, in accordance with the requirement of ISO 12106. The testpieces were fatigue loaded with a constant total strain amplitude of ±0.7% and strain rate of 10^{-3} 1/s at room temperature using a servo-controlled electromechanical 50kN machine. A Class-0.5 side-entry contacting extensometer with a gauge length of 15mm was attached to the testpiece parallel length to control and measure strain during cyclic loading.
Matlab-based subroutines were developed to determine, for every cycle, elastic modulus \((E)\), yield stress \((\sigma_0)\), effective stress \((\sigma_{\text{eff}})\), back stress \((\sigma_b)\), and loop shape parameter (defined as the ratio of the area contained within the stress-strain hysteresis loop to the area within the bounding rectangle with opposite corners at maximum and minimum stress, shown dashed in Figure 1). In this study, the approach for determining effective stress and back stress was adopted from Dickson’s method [2]. Figure 1 shows a scheme for the quantitative measurement of such parameters from a stress-strain hysteresis loop.

![Figure 1](image-url)

Figure 1: A scheme for the quantitative measurements of parameters representing elasto-plastic behaviour

Tested specimens were sectioned in parallel to the loading axis using a diamond cutting machine in order to prepare samples for microstructural observations. The samples were mechanically polished to produce thin plates of 0.1mm thickness which were then punched out to produce discs having a diameter of 3mm. These were marked to indicate the loading direction. The discs were then electrolytically polished using a double jet device (TenuPol5) with an electrolyte solution of acetic acid and perchloric acid to provide specimens for TEM investigation. Thereafter, samples were observed in a Philips CM30 transmission electron microscope at 300kV with a double tilt holder. The mark of the loading direction (LD) was aligned along the holder axis in order to preserve the loading direction during observations. Selected area electron diffraction and Kikuchi patterns were used to identify the grain orientation relative to the loading axis. TEM images were obtained under multi-beam diffraction conditions.

Dislocation density was measured from TEM images using a point intersection method which is explained in detail elsewhere [19]. Dislocation structure evolution of the material was determined by examining samples obtained from an as-received (solution treated) specimen, and testpieces fatigued up to 8, 30, 95, 700, 1600 and 3200 cycles, respectively, corresponding to the midpoint of the hardening stage, the maximum peak stress, the midpoint of the softening stage, the beginning of the stabilised stage, the midlife and the end of life (Figure 2a).

3. Results and Discussion

The cyclic deformation response of the material for ±0.7% \(\varepsilon_a\) at 22°C in terms of the maximum stress versus number of cycles is shown in Figure 2a. The mechanical response is characterised by three stages: firstly cyclic hardening, followed by cyclic softening, and finally by an almost stable response before
testpiece failure. The cyclic hardening stage occupies ~30 cycles, followed by the cyclic softening stage until about the 400th cycle. Afterwards, a stabilised response stage (saturation stage) endures until failure.

The evolution of the microstructure of the material during cyclic loading is summarised in context with the cyclic deformation response in Figure 2a. In the as-received condition, dislocations are mainly present in the form of planar structures, i.e. regular arrays of dislocations, stacking faults etc. During cyclic hardening, dislocation density increases, resulting in dense dislocation sheets with some interconnections between them at the end of this stage. Nevertheless, dislocations are still present in the forms of planar structures during the hardening stage. Dislocation density increases during the cyclic hardening stage from about $6.5 \times 10^{13} \text{ 1/m}^2$ in the as-received condition, to about $2.4 \times 10^{14} \text{ 1/m}^2$ at the end of the hardening stage. Although dislocation density is believed to be constant after the hardening peak, dislocations rearrange during the softening stage due to the activation of cross-slip and secondary slip [11,12], resulting in the formation of dislocation walls/channels. Upon further loading, wall/channel structures develop into a well-organised cellular structure. The activation of secondary slip systems creates more connections between cell walls, resulting in a labyrinth structure. Consequently, cellular structures become more equiaxed. Equiaxed cells are observed more frequently onwards from the middle of fatigue life. Persistent slip bands (PSBs) which are mainly responsible for the formation of microcracks during fatigue are found to be vigorously active during the saturation stage (Figure 2b).

During the early cycles, the shape of the stress-strain hysteresis loop becomes more pointed, indicating an increase of hardening coefficient due to the increase in dislocation density in the cyclic hardening stage. During the softening stage, the loop shape slightly changes back to be more rectangular. Correspondingly, the magnitude of the loop shape parameter decreases in value during cyclic hardening and slightly increases during softening (Figure 3a). The loop shape parameter decreases again after around 500 cycles, and in a prominent way after ~1000 cycles which is coincident with a considerable divergence between elastic moduli measured during tensile going and compressive going branches (Figure 3b). The difference between tensile going elastic modulus and compressive going elastic modulus is normally believed to be due to the formation of microcracks which are usually the consequence of PSB formation during cyclic loading. The strong activation of PSBs during the saturation stage is therefore most probably responsible for the observed decrease in loop shape parameter.
The evidence in Figure 4a indicates that the cyclic hardening stage is due to increases in both effective stress and back stress. Since dislocation density increases during the first cycles, effective stress increases due to the forest hardening mechanism. In addition, the formation of dislocation pile ups and differences in the distribution of dislocations in areas close to grain boundaries and in the middle of grains (Figure 4b) are believed to be responsible for the significant increase in back stress during the cyclic hardening stage. After the hardening peak, while the effective stress is almost constant until failure, the back stress slightly decreases during the cyclic softening stage and then keeps constant during the saturation stage. The back stress can decompose into two types which are inter-granular back stress and intra-granular back stress. The inter-granular component, acting over a length scale from sub-domain to sub-domain, associates with plastic deformation incompatibilities between different grains and different sub-domains. The intra-granular term associates with the formation of dislocation-rich and dislocation-poor structures (i.e. walls and channels). The activation of secondary slip systems and homogenisation of the dislocation distribution at the inter-granular scale is responsible for the slight decrease in the inter-granular back stress. However, during the cyclic softening stage, the formation of dislocation veins/walls causes increase in the intra-granular back stress which compensates for the decrease in the inter-granular back stress.
stress. Thereafter, the back stress reaches a stable value once well-organised dislocation structures form which is the case during the saturation stage.

4. Conclusion

The cyclic deformation response of AISI 316L steel is characterised in terms of its tensile peak stress response during strain controlled low cycle fatigue by three stages: a hardening stage followed by a softening stage, and finally a saturation stage. The analysis of stress-strain hysteresis loops shows that the hardening stage is due to an increase in both effective stress and back stress which is associated with the evolution of dislocation density and the distribution of the dislocations. During the softening stage, while effective stress is constant after the hardening peak, the inter-granular component of back stress decreases as a consequence of the activation of secondary slip, but is compensated by an increase in the intra-granular component of back stress due to the formation of dislocation walls/channels. Finally, back stress obtains its stable value as dislocation well-organised structures form late in the softening stage. Analyses of the loop shape parameter and the elastic moduli of tensile going and compressive going loop branches provide an indication of microstructural evolution, in particular the activation of PSBs which are responsible for the formation of microcracks during cyclic loading.

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References

[1] D. Kuhlmann-Wilsdorf and C. Laird, Mater. Sci. & Eng. 37 (1979).
[2] J. I. Dickson, J. Bountin and L. Handfield, Mater. Sci. & Eng. 64 (1984).
[3] X. Feaugas, Acta Metall. 47 (1999).
[4] X. Feaugas and C. Gaudin, Mater. Sci. & Eng. A309-310 (2001).
[5] H. Mughrabi, Mater Sci Eng 33 (1978) 207-223.
[6] J. Polak, F. Fardoun and S. Degallaix, Mat Sci Eng a-Struct 215 (1996) 104-112.
[7] A. Abel and Y. S. Chung, Scripta Metall Mater 13 (1979) 907-910.
[8] H. Mughrabi, Acta Metall. 31 (1983).
[9] M. Gerland, J. Mendez, P. Violan and B. A. Saadi, Mat Sci Eng A 118 (1989) 83-95.
[10] N. Y. Jin, C. H. Zhong and X. F. Chen, Acta Metall Mater 38 (1990) 2141-2148.
[11] Y. F. Li and C. Laird, Mat Sci Eng A 186 (1994) 65-86.
[12] Y. F. Li and C. Laird, Mat Sci Eng A 186 (1994) 87-103.
[13] T. Kruml, K. Obtrlik and J. Polak, Kovove Mater 31 (1993) 529-540.
[14] K. Obtrlik, T. Kruml and J. Polak, Mat Sci Eng A 187 (1994) 1-9.
[15] J. Polak, K. Obtrlik and M. Hajek, Fatigue Fract Eng M 17 (1994) 773-782.
[16] H. Mughrabi and H. J. Christ, Isij Int 37 (1997) 1154-1169.
[17] D. Y. Ye, S. Matsuoka, N. Nagashima and N. Suzuki, Mat Sci Eng A 415 (2006) 104-117.
[18] T. Mayama, K. Sasaki and M. Kuroda, Acta Mater 56 (2008) 2735-2743.
[19] M. S. Pham, C. Solenthaler, K. G. F. Janssens and S. R. Holdsworth, Mat Sci Eng A 528 (2011) 3261-3269.