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Fatigue damage of AISI 304 LN stainless steel: Role of mean stress

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Abstract

This investigation examines the influence of varied combinations of mean stress (\(\sigma_m\)) and alternating stress (\(\sigma_a\)) on strain accumulation during fatigue tests and the resultant in-situ microstructural and substructural variations of AISI 304 LN stainless steel. Stress-controlled fatigue tests have been carried out at 300 K using positive, zero, and negative mean stress conditions, supplemented by microstructural and substructural analyses using TEM and XRD. The results highlight that the characteristics of strain accumulation in the selected steel is governed by the magnitude of \(\sigma_m\) and \(\sigma_a\), apart from considerable dependence on in-situ formation of deformation induced martensite and substructural changes.

Keywords: Mean stress; stress amplitude; strain accumulation; deformation induced martensite; AISI 304LN stainless steel.

1. Introduction

The damage of structural components under cyclic loading is a century-old well known engineering problem but significant emphasis is still being laid on this domain of research. Cyclic loading behaviour of structural materials is commonly dealt with low cycle fatigue (LCF), high cycle fatigue (HCF) and fatigue crack growth rate (FCGR). One of the current problems related to LCF is to understand the influence of symmetric (mean stress = 0) vis-a-vis asymmetric (mean stress \(\neq 0\)) loading conditions. The primary driving force to achieve this understanding is: imposition of asymmetric stress cycle leads to enhanced strain accumulation in an engineering component [1-3]. Accumulation of this type of additional plastic strain decreases fatigue life [4, 5] and limits the predictive capability of the Coffin-Manson relation [6]. This is one of the serious issues in critical engineering structures such as in nuclear power plants, since the accumulated plastic strain governs the life of engineering components subjected to LCF. Thus one should critically examine the influence of strain accumulation in engineering components in order to safe guard these against both symmetric and asymmetric loading conditions. Literature pertinent to this discipline provides information related to strain accumulation behaviour under uniaxial or multiaxial loading conditions for cyclic hardening and cyclic softening type metallic materials [7-9]; but the existing information and the generated knowledge is still insufficient to crystallize our concepts to make comparative assessment of strain accumulation...
under symmetric and asymmetric loading conditions. In particular, most of the earlier investigations related to strain accumulation addresses the mechanics of materials; but micro-mechanisms such as possible effect of in-situ substructural and microstructural changes leading to plastic strain accumulation during asymmetric cyclic tests has not been carefully dealt with in the earlier investigations. The primary aim of this report concerns this aspect related to austenitic stainless steels.

A series of investigators like Nagy et al. [10], De et al. [11], Smaga et al. [12], Krupp et al. [13] and Das et al. [14] etc. have shown that austenitic stainless steels are unstable upon monotonic or cyclic deformation, which readily exhibit transformation of austenite to martensite. The martensitic transformation occurs when austenitic stainless steels are deformed at temperatures below \( M_d \), a temperature below which deformation induced martensitic transformation takes place [14, 15]; typically the value of \( M_d \) could be 275 K [14]. Thus, formation of martensite even at room temperature is not ruled out in 304 type stainless steels. It is already known that strain accumulations during symmetric and asymmetric loading are different and the extent of martensitic transformation depends up on the extent of deformation. Hence, it is naturally expected that the structural changes during LCF of 304LN stainless steel would be different during LCF associated with symmetric or asymmetric loading conditions. In addition, the substructural changes occurring under these two conditions of loading would be different from the point of view of fatigue mechanics as well as due to the variation in the structural changes occurring during cyclic loading. The aim of this investigation is to examine the nature of strain accumulation in AISI 304LN stainless steel through stress-controlled fatigue tests, at various combinations of mean stress and stress amplitude; this is supplemented with examinations on structural and microstructural changes using XRD and TEM.

### Nomenclature

- \( \sigma_m \) mean stress
- \( \sigma_a \) stress amplitude
- \( \varepsilon_p \) strain accumulation
- \( \varepsilon_{pf} \) total strain accumulation at failure
- \( N \) number of cycles
- \( N_f \) total number of cycles at failure

### 2. Experimental

The chemical composition of the selected AISI 304LN stainless steel is Fe–0.03C–18.55Cr–9.50Ni–0.54Si–0.028P–0.014S–0.1N–1.80Mn (all in wt.%). Initially large pieces were cut from a pipe of 320 mm outer diameter and 25 mm wall thickness. Sample blanks with approximate cross-sections of 10 mm x 10 mm were cut from the as-received material for metallographic examinations and hardness measurements. The microstructural examinations were carried out with the help of an optical microscope (Leica, model: DMILP, USA) which was attached to an image analyzer (Software: Biovis Material Plus, Version: 1.50, India). The hardness measurements were done with the help of a Vickers hardness tester (Leco, model: LV 700, USA) using an indentation load of 20 kgf. Tensile specimens were prepared as per ASTM standards [16]. The tensile tests were carried out on cylindrical specimens of 6 mm diameter and 25 mm gauge length (Fig. 1a) at a nominal strain rate of \( 1 \times 10^{-3} \) s\(^{-1} \) at room temperature (300 K).

Specimens for stress-controlled fatigue tests were fabricated from the pipe in such a manner that the loading axis coincides with the pipe axis. The specimens were cylindrical with 7 mm gauge diameter and 13 mm in gauge length (Fig. 1b). Stress-controlled fatigue experiments were carried out at 300 K using 100 kN servo-electric testing system (Instron, model: 8862, UK). All controls and data acquisition were computerized. Tests were done in stress-control mode till fracture using triangular waveform at a constant stress rate of 50MPa/s. The variables that have been considered for these tests are mean stress \( (\sigma_m) \), and stress amplitude \( (\sigma_a) \). The combinations of the adopted \( \sigma_a \) and \( \sigma_m \) for each test are listed in Table 1. Based on the employed test controls, the tests can be classified into four categories: (i) constant \( \sigma_a \) with varying \( \sigma_m \), (ii) constant \( \sigma_m \) with varying \( \sigma_a \), (iii) \( \sigma_m = 0 \) and (iv) negative \( \sigma_m \). It may
be noted that due to the wide selected range of $\sigma_m$ (positive, zero and negative), the specimens have been subjected to deformation under varying tension-compression cycles. The strain measurements during cyclic deformation were made using an axial extensometer having 12.5 mm gauge length. During each test, the stress-extension as well as the actuator displacement data were continuously recorded with an aim to acquire at least 200 data points per cycle for further analyses.

Transverse sections from the gauge portion of the specimens were cut after the fatigue tests and were subjected to XRD analyses using Cu Kα radiation. These analyses were made at a scan rate of 1.5° per minute using a high resolution X-ray diffractometer (Philips, model: PW1710, UK). In addition, thin slices (~0.5 mm thick) were also cut from the gauge section of the tested samples for preparing TEM samples using a slow speed saw (Buehler, USA). The slices were then thinned sequentially by manual polishing on emery paper, dimpled and ion milled prior to TEM studies. TEM studies were carried out using a 200 keV transmission electron microscope (JEOL, model: JEM 2100, Japan).

| Sl. No. | $\sigma_m$ (MPa) | $\sigma_s$ (MPa) |
|--------|-----------------|-----------------|
| 1      | -60             | 420             |
| 2      | 0               | 420             |
| 3      | 50              | 300, 350, 400   |
| 4      | 100             | 300, 350, 400   |
| 5      | 150             | 300, 350, 400   |

3. Result and discussion

The characteristics of the microstructural features and conventional mechanical properties are described in section 3.1 while the strain accumulation behaviour of the material is discussed in section 3.2 to section 3.4 and substructural and microstructural variations is discussed in section 3.5.

3.1. Microstructure and mechanical properties

The microstructure of the as received AISI 304LN stainless steel is illustrated in Fig. 2 which exhibits nearly equiaxed austenite grains in association with annealing twins. The average grain size of the steel is estimated as 65±4.3 μm and its Vickers hardness (HV20) is 205. The average tensile properties of the steel can be summarized as: yield strength 340 MPa, tensile strength: 683 MPa and percentage elongation: 70.6 %.
3.2. Nature of strain accumulation: Effect of symmetric cyclic loading (i.e. $\sigma_m = 0$)

Typical hysteresis loops generated from the fatigue tests carried out under symmetric loading are shown in Fig. 3a and the corresponding strain accumulation behaviour with increasing number of cycles is shown in Fig. 3b. The results indicate that total strain accumulation is insignificant when $\sigma_m = 0$ at any level of $\sigma_a$. In this case $\sigma_a = 420$ MPa, but only a maximum of 6% strain is accumulated and it decreases for decreasing $\sigma_a$. The results are in accordance with some earlier investigations where it has been reported that no significant strain accumulation occurs under symmetric loading. This phenomenon can be described using the dislocation substructure that forms during cyclic tests with zero mean stress. The number of dislocations those generate during the forward cycle are comparable with those generated during the backward cycle, and hence there is a possibility of a considerable part to get annihilated. This fact reduces strain accumulation in symmetric cyclic loading.

3.3. Nature of strain accumulation: Effect of asymmetric cyclic loading

The results of cyclic tests conducted under different combinations of mean stress and stress amplitude are discussed in this section. The mean stress is varied from positive to negative.
3.3.1. Effect of stress amplitude at constant positive mean stress

The variations of strain accumulation ($\varepsilon_p$) with number of cycles (N) for varying $V_a$ at constant $V_m$ levels of 50, 100 and 150 MPa were examined and a typical illustration is shown in Fig. 4a. The results indicate that $\varepsilon_p$ increases monotonically with increasing N for any $V_a$ - $V_m$ combination. In addition, the magnitude of $\varepsilon_p$ increases with increasing $V_a$ at a constant $V_m$ and at any specific cycle. The variations in total accumulated strain ($\varepsilon_{pf}$) at failure (i.e. N=Nf) with increasing level of $V_m$ at different $V_a$ values are summarized in Fig. 4b; the results in this figure illustrate increase in $\varepsilon_{pf}$ with increasing level of $V_m$. When $V_a$ is altered between 300 and 400 MPa, the magnitudes of $\varepsilon_{pf}$ increase from 7.2 to 24.2 % at $V_m = 50$ MPa, from 7.7 to 39.5 % at $V_m = 100$ MPa and from 14.6 to 73.6 % at $V_m = 150$ MPa. The observed nature of increase in $\varepsilon_p$ with $V_a$ is in good agreement with the trend of results reported by Kang et al. [17] in SS304 stainless steel and that by Gupta et al. [18] in SA333 Gr. 6 piping steel.

The results presented in Fig. 4a infer that for a given $V_m$ level, fatigue life ($N_f$) of the material decreases with increasing $\varepsilon_{pf}$. The decrease in $N_f$ with increasing $V_a$ for various $V_m$ values is compiled in Fig. 5. For example, for the combination of $V_m = 150$ MPa and $V_a = 300$ MPa, failure of a specimen occurs at 12661 cycles but for the combination of $V_m = 150$ MPa and $V_a = 400$ MPa, its fatigue life reduces to 1403 cycles (Fig. 4). Kang et al. [17] have shown that for the combination of $V_m = 65$ MPa and $V_a = 325$ MPa, failure occurs after 3000 cycles for solution annealed SS 304 steel; for nearly similar experimental conditions of $V_m = 50$ MPa and $V_a = 300$ MPa, the investigated 304LN stainless steel exhibits $N_f = 6280$ cycles. The difference in the observation can be attributed to the difference in the pre-history of the materials used in the two investigations.

This phenomenon of strain accumulation at constant $V_m$ for varying $V_a$ can be correlated with the dislocation substructure of the material. Due to the selected nature of the loading-unloading cycles, the number of dislocations generated in the loading cycle is more than that generated in the unloading cycle; but, during load reversal, only a part of the dislocations get annihilated. As a result some amount of the generated dislocations would remain as residuals in the substructure of the investigated steel subjected to asymmetric cyclic loading. It is well known that higher is the dislocation density, higher is the accumulation of plastic strain and vice versa. Hence, it may be inferred that with increasing $V_a$ for a particular $V_m$, total strain accumulation will increase because of the increase in the remnant dislocation density.
3.3.2. Effect of mean stress at constant stress amplitude ($V_m>0$)

To understand the effect of $V_m$ on $H_p$ at constant $V_a$ levels, the results presented in the previous section have been re-examined as $H_p$ vs. $N$ at constant $V_a$. The observed behaviour is illustrated for all the investigated $V_a$ levels in Fig. 6. The increase in $H_p$ due to increase in $V_m$ at constant $V_a$ has been discussed in the previous section. The relative increase of fatigue life with increasing $V_m$ can be explained in the following manner. At constant stress amplitude, if $V_m$ is increased, the magnitude of $V_{min}$ decreases, as a result, the hysteresis loop move upward. For example, at $V_a = 300$ MPa when the magnitudes of $V_m$ are 50 and 100 MPa, that of $V_{min}$ are -250 and -200 MPa respectively. This phenomenon results in shifting the cyclic deformation zone more towards the tension-tension side and, as a consequence the extent of reverse plastic deformation during asymmetric cyclic loading is lower in a specimen.

3.3.3. Effect of stress amplitude at constant negative mean stress

It is established from the earlier discussions that mean stress has the most significant role in controlling strain accumulation during asymmetric cyclic loading. As remnant dislocations exist after the completion of each cycle, which contributes to strain accumulation, it is necessary to examine the strain accumulation behaviour by cycling for longer duration in the compression side, and this condition can be achieved by applying negative mean stress. Typical hysteresis loops generated from fatigue tests carried out under asymmetric cyclic loading with negative mean stress are shown in Fig. 7a, and the corresponding strain accumulation behaviour with increasing number of cycles is shown in Fig. 7b. The results indicate that the nature of strain accumulation is almost similar with that of test conditions with positive mean stress, except that the direction of strain accumulation is just opposite in comparison to the cases involving positive mean stress. Here also, the total strain accumulation increases with the increase in $V_a$ at a constant $V_m$ level.

![Fig. 6. Histograms showing variations of total accumulated strain ($\varepsilon_{pf}$) and $N_f$ with $\sigma_a$.](image)

To understand the effect of $\sigma_m$ on $\varepsilon_{pf}$ at constant $\sigma_a$ levels, the results presented in the previous section have been re-examined as $\varepsilon_{pf}$ vs. $N$ at constant $\sigma_a$. The observed behaviour is illustrated for all the investigated $\sigma_a$ levels in Fig. 6. The increase in $\varepsilon_{pf}$ due to increase in $\sigma_m$ at constant $\sigma_a$ has been discussed in the previous section. The relative increase of fatigue life with increasing $\sigma_m$ can be explained in the following manner. At constant stress amplitude, if $\sigma_m$ is increased, the magnitude of $\sigma_{min}$ decreases, as a result, the hysteresis loop move upward. For example, at $\sigma_a = 300$ MPa when the magnitudes of $\sigma_m$ are 50 and 100 MPa, that of $\sigma_{min}$ are -250 and -200 MPa respectively. This phenomenon results in shifting the cyclic deformation zone more towards the tension-tension side and, as a consequence the extent of reverse plastic deformation during asymmetric cyclic loading is lower in a specimen.

![Fig. 7. Typical (a) hysteresis loops generated during asymmetric cyclic loading with negative mean stress (all the cycles are not shown in the figure) and (b) variation of strain accumulation with number of cycles at a loading condition of $\sigma_m = -60$ MPa, and $\sigma_a = 420$ MPa.](image)
3.4. Saturation in strain accumulation

The variation in the rate of strain accumulation (RSA) with increasing number of cycles for the condition of “constant \( \sigma_\text{a} \)-varying \( \sigma_\text{m} \)” is presented in Fig. 8. The results indicate that \( \frac{dH_p}{dN} \) (or RSA) decreases with increasing number of cycles in any combination of \( \sigma_\text{m} \) and \( \sigma_\text{a} \). Similar results were observed for “constant \( \sigma_\text{m} \)-varying \( \sigma_\text{a} \)” conditions. The decrease in RSA is rapid up to about ten cycles, after which the change in the magnitude of RSA is insignificant. The magnitude of RSA becomes almost constant after about 100 cycles for all combinations of \( \sigma_\text{m} \) and \( \sigma_\text{a} \). In brief, it can be inferred that rapid accumulation of strain in the initial few cycles followed by attainment of a steady state in RSA are the characteristic of the asymmetric cyclic deformation behaviour of 304LN stainless steel. The observed results infer that the change in strain accumulation is sharp in the initial cycles. This observation could possibly be attributed to the larger amount of martensitic transformation in the cycles; the martensitic transformation is known to be associated with generation of higher amount of dislocations. The attainment of steady state in RSA can be explained by the formation, movement and redistribution of dislocations associated with cyclic deformation. When the steel is subjected to cyclic deformation, new dislocations generate and results in strain hardening. These dislocations initially form tangles and subsequently lead to the formation of dislocation cells with increasing number of cycles [3]. After certain number of cycling, depending on the magnitude of the imposed cyclic strain, the newly generated dislocations assume a stable configuration and this leads to the steady state in RSA.

3.5. In-Situ variation of substructure and microstructure

To understand the substructural and microstructural variations associated with the deformations under asymmetric cyclic loading of the investigated 304 LN stainless steel, the deformed specimens were examined using TEM and X-ray diffraction studies. Typical TEM bright field images obtained from thin foils cut from the transverse sections of deformed specimens are illustrated in Fig. 9. Comparison of the substructural features in Fig. 9a and 9b leads to infer that dislocation density increases with increase in the magnitude of \( \sigma_\text{m} \).

Gaudin and Feaugas [3] have reported that strain accumulation under asymmetric loading results in the formation and dissolution of dislocation cells during cycling. Dislocation cell formation is also evident from the TEM results of the present investigation.

In the as received condition, the microstructure of the investigated 304 LN stainless steel was completely austenitic. However, as a result of monotonic or cyclic deformation, this grade of stainless steel has been reported to show phase transformation from fcc austenite to metastable \( \epsilon \)-martensite and eventually to \( \alpha' \)-martensite [11]. TEM results unambiguously indicate that martensite forms during the asymmetric cyclic deformation. A typical TEM bright field image along with the corresponding selected area diffraction pattern is
shown in Fig. 10 to provide evidence for the formation of martensite during cyclic deformation of this steel. X-ray diffraction analyses show that both $\varepsilon$-martensite (hcp) and $\alpha'$-martensite (bcc) peaks are present in the diffraction pattern (Fig. 11).

![Fig. 10. Formation of $\alpha'$-martensite (bcc) (directed by arrow markings) with zone axis of $[\bar{1}13]$ in $\gamma$-austenite (fcc) with zone axis of $[-2\bar{3}3]$.](image1)

In generalization, three synergistic phenomena are taking place during asymmetric cyclic deformation of AISI 304 LN stainless steel: (i) variation in dislocation density, (ii) formation, followed by dissolution or growth or shrinkage of the dislocation cells, and (iii) martensitic transformation. It is noted that the deformation induced martensite can be stable $\alpha'$ or the metastable $\varepsilon$ and their role in altering the remnant dislocation density or the cyclic deformation process in influencing $\varepsilon_p$ or $N_f$ remains unclear at this stage. However, it can be unambiguously concluded that $\sigma_m$ has more significant effect than $\sigma_a$ in controlling strain accumulation behaviour of the investigated 304 LN stainless steel.

4. Conclusions

A series of fatigue experiments have been carried out on 304LN stainless steel using stress control mode with a number of combinations of mean stress and stress amplitude. The results obtained from these experiments have been critically analyzed which lead to the following inferences/conclusions:

a) The extent of strain accumulation in the selected AISI 304LN stainless steel increases with increase in the magnitude of peak stress of the imposed cyclic loading, when the magnitude of mean stress is kept constant at pre-selected maximum stress values encountered in a cycle. The magnitude of strain accumulation is found to be marginal when tests are carried out under symmetric cyclic loading; but its magnitude is considerable when the tests are done under asymmetric loading whether the mean stress is positive or negative.

b) When the magnitude of the mean stress is kept constant, the number of cycles to cause fatigue failure decreases with increase in the amplitude of alternating stress; whereas the number of cycles to cause fatigue failure increases with increase in the imposed magnitude of mean stress at constant alternating stress.

c) Approximate saturation of strain accumulation occurs at $\geq 100$ cycles of loading for AISI 304 LN stainless steel for all the selected test conditions.

d) TEM studies lead to infer that dislocation density of the cyclically deformed samples increases with increasing $\sigma_m$ at constant $\sigma_a$. X-ray diffraction studies indicate that deformation induced martensite forms during asymmetric cyclic loading. This type of martensitic transformation also adds up new dislocations in the deformed samples. The increase or decrease of strain accumulation under asymmetric loading has been explained with the considerations of remnant dislocation density, reverse plastic deformation and shifting of the deformation zone.
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