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# Low cycle fatigue and creep-fatigue interaction behaviour of 316L(N) stainless steel and its welds

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#### Abstract

High temperature low cycle fatigue (LCF) is influenced by various time dependent processes such as creep, oxidation, phase transformations and dynamic strain ageing (DSA) depending on test conditions of strain rate and temperature. In this paper the detrimental effects of DSA and oxidation in high temperature LCF are discussed with reference to extensive studies on 316L(N) stainless steel. DSA has been found to enhance the stress response and reduce ductility. It localizes fatigue deformation, enhances fatigue cracking and reduces fatigue life. High temperature oxidation accelerates transgranular and intergranular fatigue cracking during long hold time tests in austenitic stainless steel. In welds, microstructural features such as presence of coarse grains and formation of brittle phases due to transformation of  $\delta$  ferrite during testing influence crack initiation, propagation and fatigue life.

#### 1. Introduction

Low Cycle Fatigue (LCF) is an important consideration in the design of high temperature systems subjected to thermal transients. The systems that experience thermal transients include aircraft gas turbines, nuclear reactor vessels, heat exchangers, steam turbines and other power plant components. LCF resulting from thermal transients occurs essentially under strain controlled conditions, since the surface region is constrained by the bulk of the component. In thick components the major compressive strain is introduced by the thermal transient during start up, with additional compressive and or tensile strains during load cycling and shut down. On-load periods at elevated temperatures in between transients introduce time-dependent effects. Temperature time transients which could be experienced by components in a fast reactor are the result of reactor trip (down shock) or secondary circuit failure (up shock) [1].

Traditionally, isothermal low cycle fatigue tests have been used to assess the performance of materials subjected to thermal transients. Thus the component behaviour is studied using mechanical strain cycling under isothermal testing conditions. The slow start up/shut down cycle is replaced by a symmetrical and continuous fatigue cycle of equal strain rates in tension and compression with a hold period at constant peak strain to simulate the on-load period.

At high temperatures the fatigue deformation and life are influenced by several time dependent mechanisms such as dynamic strain ageing (DSA), oxidation, creep and phase transformations. These damage processes which are strong functions of temperature and strain rate, are illustrated with examples from extensive studies conducted on 316L(N) stainless steel and their welds [2-8] which are the currently favoured structural materials for the primary side of the liquid metal cooled fast reactor.

Nitrogen modified 316L(N) austenitic stainless steel is used in nuclear power plants for the construction of reactor vessel, piping and heat exchangers. Evaluation of elevated temperature LCF behaviour of 316L(N) stainless steel has received much attention in the recent years [2,9-15]. In these studies, nitrogen addition has been reported to be beneficial, and the LCF life has been found to improve at both ambient temperature and 873 K.

#### 2. Experimental

The 316L(N) base metal in the mill-annealed condition was solution treated at 1373 K for 1 hour, followed by water quenching prior to machining the LCF specimens. Weld metal specimens were machined from weld pads prepared by shielded metal arc welding process using 316 and 316(N) electrodes. X-ray radiography was used for assessing the soundness of the welds followed by  $\delta$ -ferrite measurements using a magne-gauge

#### 2.1 Low cycle fatigue testing

Fully reversed total axial strain controlled LCF tests were conducted at 773, 823 and 873 K in air on the 316L(N) base metal, 316 weld metal and 316(N) weld metal specimens using a servo hydraulic machine equipped with a radiant heating facility. Tests were carried out with total strain amplitudes in the range  $\pm 0.25\%$  to  $\pm 1.0\%$  with the strain rates varying from  $3\times10^{-5}$  s<sup>-1</sup> to  $3\times10^{-2}$  s<sup>-1</sup>. Creep-fatigue interaction experiments were conducted by introducing tension/ compression hold in the range 1 min. to 90 min. at 873 K.

#### 2.2 Metallography

The tested samples were sectioned parallel to the loading direction, polished, etched and examined under an optical microscope. The 316L(N) base metal was etched using 70% HNO<sub>3</sub> while the etching of weld metal was done using a modified Murakami's reagent (30g. of KOH, 30g. of  $K_3Fe(CN)_6$  in 150 ml water) at 363 K for 30 seconds. Fractography of the failed specimens was carried out using a PSEM 501 scanning electron microscope and substructural changes were studied by Philips CM 12 transmission electron microscopy (TEM) were obtained from thin slices cut at a distance of 3 mm away from the fracture surface. These samples were first mechanically polished down to 250mm, and then electropolished in a solution containing 20% perchloric acid and 80% methanol at 243 K with a d.c. voltage of 10 V.

#### 3. Results and discussion

## 3.1 LCF of 316L(N) Stainless Steel: Cyclic Properties and Role of DSA

High temperature LCF behaviour of 316L(N) stainless steel has been studied by several investigators [2-8]. The dependence of the peak tensile stress on the number of cycles and on total strain amplitude for the base metal at 773 K is depicted in Fig. 1a. This material generally exhibits a very rapid strain hardening to a maximum stress followed by a nearly stable peak stress. The initial hardening observed in the base metal is mainly due to dynamic strain ageing caused by the interaction between dislocations and solute atoms. DSA was found to manifest in the cyclic stress response of 316L(N) in the temperature range 773-873 K, Fig. 1(b)(Temperature 823K), where a negative strain rate dependence of stress response is observed. The negative strain rate-stress response observed at these temperatures and strain rates was also reflected in a negative strain rate dependence of half-life stress [8]. Further, at all these temperatures, at strain rates  $< 3 \times 10^{-3}$  s<sup>-1</sup>, servated flow was observed (Fig. 1(c)) and the cyclic stress response increased with increasing temperature in the range 573- 873K[8].

In the DSA regime, enhanced slip planarity and the degree of inhomogeneity of deformation is observed [8]. Further, the dislocation structure changed under the influence of increasing contribution from DSA. The solute dislocation interaction during DSA restricted cross slip of dislocations and increased the slip planarity. Dislocation structure changes from a cell structure at temperatures below 573 K, to planar one between 573 and 873 K (DSA regime), and back to a cell/ subgrain microstructure beyond the DSA regime[8]. Planar slip bands were observed in this material in the temperature and strain rate regime where DSA was predominant [8]. This exerted a profound influence on the fatigue life.

In order to assess the influence of planar slip on cracking, the total length of intergranular and transgranular crack density were measured on the specimen surface in the DSA regime. Crack density (crack length per unit area) as a function of strain rate at 773 K is shown in Fig. 2(a) and the number of cracks (of different crack length) as a function of strain rate at 773 K is shown in Fig. 2(b). It is noticed that in the DSA regime, both transgranular and intergranular crack density increases with decrease in strain rate (Fig. 2a) and very long cracks are seen (a consequence of crack coalescence) at low strain rates. The stress concentration associated with the intersection of planar slip bands with the



Fig. 1 : (a) Cyclic stress response – strain amplitude effect; (b) Cyclic stress response – strain rate effect; (c) Serrations on the stress – strain hysteresis loops



Fig. 2 : (a) Crack density as a function of strain rate; (b) Crack length distribution as a function of strain rate

grain boundaries could have contributed to the enhanced internal grain boundary cracks and reduced lives at low strain rates, Fig. 3. The continuous decrease in fatigue life with a decrease in strain rate and increase in temperature in the base metal is thus attributed to DSA under conditions where the effects of oxidation and creep are non - existent [8].

The negative strain rate dependence of cyclic stress response over the temperature and strain rate range where DSA operates results from an increase in total dislocation density during deformation. The matrix was hardened during DSA, causing an increase in flow stress needed to impose the same total strain during successive cycles.

## 3.2 Role of microstructure and phase transformation in LCF of stainless steel welds

Low cycle fatigue behaviour is affected by the initial microstructure and the microstructural changes that occur during deformation at high temperature. The dependence of the peak tensile stress on the number of cycles and on total strain amplitude for 316 weld metal, at 773 K and 316L(N)/316 weld joint at 873 K is depicted in Figs. 4a-b. Weld metal undergoes a relatively short initial hardening followed by a continuous softening regime without any apparent saturation period (Fig.4 a). The weld joints also displayed an initial hardening followed by a softening regime at all strain



Fig. 3 : Fatigue life as a function of strain rate



Fig. 4 : Cyclic stress response of (a) 316 weld metal, 773 K and (b) 316L(N)/316 weld joint at 873K



Fig. 5 : (a) High dislocation density in austenite matrix before testing; (b) Low dislocation density in austenite matrix after LCF testing

amplitudes, except at low amplitudes where a saturation stage was observed (Fig. 4b).

In order to elucidate the operative deformation mechanisms detailed transmission electron microscopy of 316 weld metal was undertaken [4]. Untested samples revealed a very high dislocation density (Fig. 5a) and the dislocation structure was mainly tangles. The configuration of dislocations in the austenite matrix of the weld metal in the untested condition resembled that of a highly cold-worked structure. This high dislocation density resulted from shrinkage stresses present during cooling of the weld metal during welding operations.

No subgrains/cells were observed in the weld metal after fatigue testing. However, dislocation density after testing was much lower compared to as welded material, Fig. 5(b). This reduction in dislocation density could result from cyclic deformation which led to break down of dislocation tangles and subsequent annihilation of the dislocations.

A comparison of LCF lives of the base metal, weld metals and weld joints at 773 K and 873 K revealed that at 773 K, the 316L(N) base metal showed a better fatigue resistance than 316 weld metal, Fig. 6(a). Further, 316(N) weld metal showed a lower fatigue endurance than 316 weld metal. However, at 873 K, 316 weld metal exhibited the highest fatigue resistance, Fig..6(b). At both the temperatures 316L(N)/316(N) weld joints showed lowest fatigue life. The variations in the fatigue life among the base metal, weld metal and weld joints with testing conditions can be correlated with the differences in the crack initiation and propagation behaviour.

Metallographic observation of fatigue tested samples revealed that, in the base metal and 316 weld metal, crack initiation occurred in purely transgranular mode at 873 K,



Fig. 6 : Fatigue life of base metal and weld metals at (a) 773 K and (b) 873 K

Fig.7a [4]. However, in weld joints, crack initiation occurs intergranularly in the HAZ (Fig. 7(b)) where grain growth has occurred during welding. The decrease in life corresponded to a transition in crack initiation mode from purely transgranular mode to an intergranular one, in the weld joint.

Fatigue crack propagation is transgranular in base metal and weld joints. In 316 weld metal, at 773 K, no crack path deflection is noticed along austenite/ferrite interface (Fig. 7(c)), unlike at 873 K, Fig.7(d). Further, in 316(N) weld metal, macrocrack propagation occurred without significant crack deflection, unlike 316 weld metal. The fine duplex austenite-ferrite microstructure in 316 weld metal with its many transformed phase boundaries at these testing conditions, offered a greater resistance to the extension of the fatigue cracks by causing deflection of the crack paths compared to the base metal.  $\delta$ -ferrite got transformed to brittle  $\sigma$  phase during testing. This transformation is found to increase with increase in temperature (773 to 873 K) and decrease in strain rate, Table 1. The extent of transformation was found to influence the fatigue life [4,7].

316(N) weld metal with higher nitrogen content (0.09%) exhibited a better fatigue resistance than the one with lower nitrogen (0.07%), Figs. 6(a) and (b). This can be rationalized based on the influence of N on fatigue deformation of austenitic stainless steels. The beneficial effects of N on LCF

Table 1. Extent of transformation of  $\delta$  ferrite in 316 and 316 (N) weld metals

Temperatures, K		773				873			
Strain amplitudes, %	0.25	$\pm 0.4$	±0.6	$\pm 1.0$	±0.25	$\pm 0.4$	±0.6	±1.0	
316 weld metal	2		_		16	25	40	40	
316(N) weld metal	_			_	10	30	30	30	



Fig. 7 : (a) Transgranular crack initiation and propagation in base metal, 873 K ; (b) Intergranular crack initiation in HAZ, weld joint, 873 K ; (c) Reduced crack deflection in 316 weld metal, 773 K ; (d) Crack deflection along the transformed d ferrite boundaries, 316 weld metal, 873 K

life of austenitic stainless steels have been studied systematically by several investigators[9-15]. In these studies, nitrogen addition has been reported to be beneficial, and the LCF life has been improved at both ambient temperature and 873 K. However, this beneficial effect of nitrogen has been found to saturate at approximately 0.12 wt.% of nitrogen. Nitrogen enhances slip planarity and the degree of reversibility of the fatigue deformation resulting in a reduced tendency for fatigue crack initiation and propagation, leading to an increased fatigue life.

#### 3.3 Effect of hold time on fatigue life

The introduction of strain hold at the peak strain in tension/compression in total strain controlled testing causes stress relaxation leading to creep-fatigue interaction. The creep-fatigue lives of the 316L(N) base metal, 316 weld metal and 316L(N)/316 weld joint as a function of the length of the hold time are shown in Fig. 8. It can be seen that, at 873 K, in both continuous cycling and hold time tests, 316 weld metal showed a higher fatigue endurance compared to the base metal. Further, the weld joint showed the lowest life. It is also evident that the hold time effect on fatigue life was



Variation of Fatigue Life with Hold Time

Fig. 8 : Fatigue life as a function of hold time



Fig. 9 : Transgranular crack propagation in 1 min. compression hold, 316L(N) base metal

dependent on the position as well as the duration of hold. Compared to continuous cycling conditions, imposition of hold at peak strain was found to decrease the fatigue life. Tensile hold was observed to be more damaging than the compression hold. Further, a significant reduction in fatigue life was observed by increasing the duration of tensile hold.

The greater creep fatigue-life of the weld metal compared to the base metal at 873 K can be correlated to the crack propagation differences between the base and weld metal. Similar to continuous cycling tests under hold time, crack deflection at the transformed  $\delta$  phase boundaries is found to increase the crack propagation resistance causing an enhanced fatigue life.

At a given strain amplitude, the relaxed stress during hold time at half life ( $\sigma_{max}$ - $\sigma_{min}$ ) represented by  $\sigma_r$  for the 316L(N) base metal is provided in Table 2. In all the hold time tests, rapid stress relaxation occurs in the first few seconds of the strain hold, followed by a slower rate of stress relaxation during the rest of the hold period. During stress relaxation, conversion of elastic to plastic strain takes place and the strain rates are typically of the order of  $10^{-4}$ s<sup>-1</sup> to  $10^{-8}$ s<sup>-1</sup> during the slow relaxation period. The build up of tensile inelastic strain leads to the accumulation of grain boundary creep damage in the form of cavities. With increase in the duration of the hold time a significant amount of stress relaxation takes place, leading to enhanced build up of intergranular creep damage. This conforms with the magnitude of  $\sigma_r$  developed during stress relaxation (Table 2) i.e.  $\sigma_r$  increases with increase in the length of the hold time



Fig. 10: Mixed mode crack propagation in 1 min. tension hold, 316L(N) base metal

and is greater in tension hold compared with the compression hold.

It must be pointed out that the absolute magnitude of the  $\sigma_r$  alone cannot be associated with the damage that determines the creep-fatigue life. As a function of the strain amplitude, it is observed that  $\sigma_r$  is relatively large at high strain amplitudes. However, the degree of reduction in life during hold time tests, defined as N/Nf (N, the fatigue life during hold-time tests and Nf the corresponding fatigue life in continuous cycling) is found to be larger at lower strain amplitudes, compared to higher strain amplitudes of testing (Table 2). The strain rates during relaxation at higher strain amplitudes are generally higher than those observed at low strain amplitudes of testing. In general, the relaxation strain rates of magnitude  $> 10^{-4}s^{-1}$  observed at high strain amplitudes correspond to those which are expected to cause marix deformation, while those observed at low strain amplitudes namely,  $\leq 10^{-4}$ s<sup>-1</sup>, correspond to that of creep deformation. It has been suggested that relaxation strain rates  $\leq 10^{-4}$ s<sup>-1</sup> generally contribute to grain boundary damage and cause a greater reduction in life [16].

The grain boundary damage developed during relaxation changes the modes of crack initiation and propagation. In 1 min. compression hold, the crack initiation and propagation occurs by transgranular mode similar to continuous cycling conditions, Fig. 9. However, under tension hold conditions crack initiation becomes intergranular and propagation is mixed mode (trans- + intergranular), Fig. 10.

Hold(min)	$\Delta \varepsilon_t / 2(\%)$	$\Delta\sigma/2(MPa)$	$\sigma_r(MPa)$	N(Cycles)	N/N <sub>f</sub>	
0	$\pm 0.6$	328	-	580(N <sub>f</sub> )	1	
1 t	"	307	51	475	0.819	
1c	"	323	45	510	0.879	
10t	"	291	57	409	0.705	
30t	"	288	74	330	0.635	
90t	"	274	94	235	0.405	
0	$\pm 1.0$	370	-	130(N <sub>f</sub> )	1	
10t	,,	342	68	140	1.08	
30t	"	322	96	110	0.846	
0	$\pm 0.4$	280	-	680(N <sub>f</sub> )	1	
10t	,,	247	40	494	0.726	

Table 2: Effect of hold time on LCF properties at 873 K



Fig. 11 : (a) Oxidized surface slip bands and (b) Oxidation induced Intergranular Cracking in 90 min. Tension Dwell Tests, 316L(N) Base Metal

It is shown in Fig. 11a that the surface-connected slip bands are oxidized under 30 min. tension hold conditions and the fracture surface is marked periodically by fatigue striations and intergranular cracks. Oxidation interaction is found to be more as the length of the hold time is increased to 90 minutes (Fig. 11b). Both crack initiation and propagation seem to be strongly assisted by oxidation and fracture surface is completely covered by a thick oxide layer.

Thus the reduced fatigue life for longer hold-times could be ascribed to the occurrence of enhanced creep and oxidation damage at grain boundaries that facilitates accelerated intergranular crack initiation and propagation, and oxidation assisted transgranular fracture.

#### 4. Conclusions

Dynamic strain ageing was found to influence cyclic hardening in 316L(N) SS in the sub-creep temperature range. The important manifestations of DSA include serrations on the stress-strain hysteresis loops, a negative strain rate-stress response and inverse temperature dependence of saturation stress. Further, enhanced slip planarity associated with DSA caused an increase in the intergranular crack density and a reduced fatigue life. Cyclic softening observed in weld metals was attributed to the annihilation of dislocations.

316L(N) base metal exhibited better fatigue resistance than 316 weld metal at 773 K. However, at 823 and 873 K, at specific strain rates, 316 weld metal showed the highest fatigue life. 316L(N) weld metal possessed lower fatigue life compared to 316 weld metal. Further, weld joints showed the lowest life under all testing conditions. Better fatigue life of 316 weld metal was attributed to a high degree of crack deflection associated with the transformed  $\delta$ -ferrite boundaries. The poor fatigue resistance of the weld joints was attributed to intergranular crack initiation and poor crack propagation resistance of the coarse-grained HAZ.

At lower strain rates and long hold times at high temperatures, oxidation was found to influence fatigue life in 316L(N) SS. The greater creep-fatigue life of the 316 weld metal compared to the base metal at 873 K under hold time testing conditions can be correlated to the crack propagation differences between the base and weld metals.

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