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Influence of nitrogen alloying on dynamic strain ageing regimes in low cycle fatigue of AISI 316LN stainless steel

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Abstract

The effect of nitrogen alloying (0.078% to 0.22%) in 316 LN stainless steel has been investigated for its Low Cycle Fatigue properties in the temperature range 300 to 873 K. The effect of dynamic strain ageing has been evaluated as a function of nitrogen content, temperature, strain amplitude and strain rate. The temperature range of operation of DSA has been found to be a strong function of the above parameters. Increase in nitrogen content shifted the range of operation of DSA to higher temperatures. For temperatures ≥ 673 K, fatigue life is found to be maximum for a nitrogen content of 0.14 wt%, while it is found to saturate or increase with increasing nitrogen content at temperatures < 673 K.

Keywords: Low Cycle Fatigue, 316 LN austenitic stainless steel, Nitrogen alloying, Dynamic Strain Ageing

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1. Introduction

316LN austenitic stainless steel with a nitrogen content of 0.09 wt% is the currently favored structural material for the primary side components (Main vessel, inner vessel, intermediate heat exchanger, etc) of Sodium Cooled Fast Reactors (SFRs). However, the section thickness of these components ranges upto 30 mm. From the view point of magnitude of thermal stresses and cost, it is necessary to reduce the section thickness and hence use high strength nitrogen-alloyed 316LN stainless steel. Nitrogen, apart from being a strong austenitic stabilizer and solid solution strengthener, introduces planar slip in the material. While cross slip promotes formation of coarse slip bands and large plastic zones at crack tip which accelerates the fatigue crack initiation and growth, planar slip induces slip reversibility that enhances the fatigue endurance by restricting the cyclic strain localisation [1]. At high nitrogen levels in austenitic stainless steels, nitrogen forms Cr-N clusters (short range order, SRO) due to strong Cr-N interactions as evidenced by Field Ion Microscope [2] and neutron spectroscopy [3]. These SROs on interaction with dislocations, promote planar slip and high stress response [4-5].

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Dynamic strain Aging (DSA) in austenitic stainless steels occur in the temperature range of 673-873 K and causes drastic reduction in Low Cycle Fatigue (LCF) life through highly localized deformation [6-7]. DSA fundamentally results from attractive interaction between solute species (or solute-atom pairs) and mobile dislocations. In the DSA region, in order to maintain an imposed strain rate, an increase in flow stress is mandatory either to unlock the dislocations from obstacles or to generate new dislocations. In austenitic stainless steels, carbon atoms are found to be responsible for DSA at low temperatures (< 350^{o}C) while nitrogen and/ or Cr-atoms at high temperatures (400-650^{o}C) [6, 8-10]. For nitrogen levels above 0.1 Wt.%, DSA has been attributed to interaction between dislocations and SROs [11]. From the above it can be inferred that, the dominance of either beneficial effect due to nitrogen addition or detrimental effect due to DSA will determine the overall fatigue endurance.

Since DSA-region encompasses SFR operating temperature (~ 820 K), it is vital to explore the manifestations of DSA and to model the LCF fatigue behavior in the DSA regime. Accordingly, exploratory tests have been designed to investigate the DSA manifestations.

2.0 Experimental Details

Chemical composition of 316LN austenitic stainless steels used in the present study is shown in Table 1.0. 316LN with nitrogen contents of 0.078 Wt. %, 0.11%, 0.14% and 0.22% are designated as N078, N11, N14 and N22 respectively. These materials are solution annealed at 1363 K / 1 h and water quenched, and resultant grain sizes lie within 85 $\pm$ 10 µm. LCF specimens with 25 mm cylindrical gauge length and 10 mm gauge diameter were used for LCF testing. To identify the temperature region of DSA at 3 x 10^{-3} s^{-1}, LCF tests were conducted in the temperature range 300-873 K at $\pm$ 0.6% total strain amplitude (mean strain = 0). In the observed DSA temperature region, effect of total strain amplitude ($\pm$ 0.4 to 1.0 %) on DSA and hence on fatigue life is examined (at 3 x 10^{-3} s^{-1}). Strain rate effect studies are conducted at 773 K at low strain rates (3 x 10^{-4} and 3 x 10^{-5} s^{-1}) to study the time dependence of DSA phenomenon. Tests were conducted in total strain control mode, in a servo-hydraulic fatigue test system, using an averaging type of extensometer. Existing results on N078 steel, from the tests conducted in our laboratory are used for comparison in these studies [7].

Table 1. Chemical Composition of nitrogen alloyed austenitic stainless steels

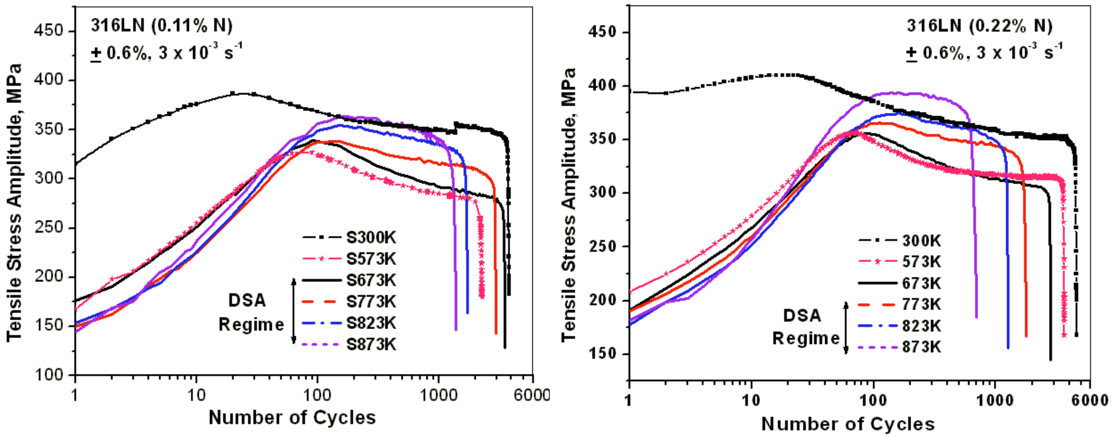
Designation	C	Cr	Ni	Mo	N	Mn	S	P
11N	0.033	17.6	12.2	2.51	0.11	1.78	0.0055	0.015
14N	0.025	17.5	12.1	2.53	0.14	1.74	0.0041	0.017
22N	0.028	17.5	12.3	2.54	0.22	1.7	0.0055	0.018
078N	0.021	17	12	2.4	0.078	1.75	0.002	0.023

3.0 Results and Discussion

In the following sections, effect of temperature, strain amplitude, strain rate and nitrogen are discussed with respect to their influence on cyclic stress response and fatigue life in the context of DSA phenomenon. Cyclic stress response represents the response of material to imposed strain cycling and is a representative of the micro-mechanisms of cyclic deformation in the material. The stress response in the present study exhibited regions of cyclic hardening, cyclic softening and cyclic saturation before the rapid stress drop concomitant with macrocrack propagation. The presence/absence and also the extent of these regions showed a strong dependence on test temperature, applied strain amplitude, strain rate and on the nitrogen content in the 316LN stainless steel. The important manifestations of DSA that can reveal its occurrence include inverse temperature and strain-rate dependence of cyclic stress, serrations in stress-strain hysteresis loops and high degree of initial cyclic hardening.

3.1 Temperature Effect

For the tests conducted in the range 300-873 K at $\pm$ 0.6% strain amplitude with strain rate of 3 x 10^{-3} s^{-1}, an anomalous stress response (i.e., increase in stress with increase in temperature) was observed in the range 673-873 K (Fig.1). This temperature range corresponds to the occurrence of Dynamic strain ageing in the material [6-7,12]. DSA essentially refers to pinning of mobile dislocations, either during dislocation glide motion [13] or during their temporary arrest at local obstacles in the glide plane [14]. This accordingly increases the resistance to dislocation motion, and hence hardens the matrix. DSA temperature regime is found to be sensitive to nitrogen content and is observed to be in the range 673-873 K (Fig.1a) for nitrogen contents $\leq$ 0.11 Wt.%, while it is shifted to slightly higher temperature range i.e., 773-873 K (Fig.1b) for nitrogen contents beyond 0.11 Wt%. The dire consequence of DSA is to cause a significant reduction in fatigue life, Fig. 2. Drastic reduction in fatigue life essentially stems from the matrix hardening that drives the crack propagation rate, as manifested by an increase in coalescence of secondary cracks and their lengths [15]. Significant amount of hardening due to DSA is clearly apparent in Fig.1, from the degree of hardening incurred in initial cyclic hardening in the DSA regime (673-873 K) compared to that in non-DSA regime (300-573 K).



(a) (b)

Fig.1: Cyclic stress response curves of 316LN austenitic stainless steel alloyed with (a) 0.11 wt.% N and (b) 0.22 wt.% N.

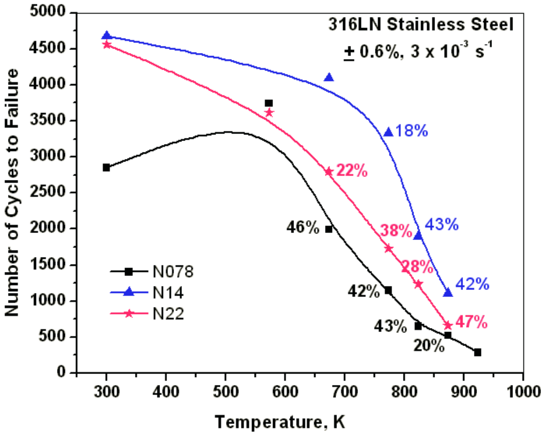


Fig.2: Influence of Nitrogen on fatigue life as a function of temperature. % values indicate the amount of reduction in life with temperature (w.r.to the life at the preceding temperature) in the DSA regime (673-873 K).

Nevertheless with increase in temperature and nitrogen content, it is possible to anticipate the contribution of precipitates to anomalous stress response, especially in case of high nitrogen levels beyond 0.11 Wt.%. It is observed that precipitation of carbides ($M_{23}C_6$ and M_6C) in 316L SS required about 50 h at 600°C [16]. Moreover, nitrogen addition to 316L SS delays the onset of carbide precipitation [2,17-18]. Therefore, under the present test conditions (i.e., test durations of < 9.5 hr at strain rates of 3 x 10^{-3} s^{-1}), contribution of carbide precipitates to matrix hardening and hence their effect on life reduction is doubtful. Absence of carbides and nitrides has also been observed in LCF tests of nitrogen-alloyed 316L SS, but however found in low strain rate ($\leq 3 \times 10^{-4}$ s^{-1}) tests at 823 K and 873 K [19].

In nitrogen alloyed 316L SS, LCF life has been found to increase with increase in nitrogen under continuous cycling conditions, but up to 0.12 wt% N only above which saturation in fatigue life has been observed in the investigated nitrogen content from around 0.03 to 0.25 wt% [11,20,21]. Increase in fatigue life has been attributed to the increased planar glide of dislocations and slip reversibility (i.e., less slip localization) as compared to low nitrogen steels [1]. Decrease in chromium diffusivity with increase in nitrogen has been experimentally determined using Atomic Force Microscopy [11] and suggested that nitrogen retards DSA by decreasing Cr-diffusivity and promotes tendency towards formation of short range order (SRO) of Cr and N. For the LCF tests (2x 10^{-3} s^{-1}, RT-600°C, $\pm$ 1.0%) on 316L SS alloyed with 0.04, 0.10, 0.13, 0.15 wt.% N [11], minimum in hardening and maximum in fatigue life are reported at 0.1% N. Based on the continuous cycling LCF tests in the present study, conducted in the temperature range 300-873 K, on 316LN with 0.11, 0.14 and 0.22 Wt% N, it is found that beneficial effect of nitrogen on fatigue life is observed to be increasing/saturating with increase in nitrogen content for temperatures < 673 K while it is found to be maximum at 0.14 wt% N at T $\geq$ 673 K. The reduction in fatigue life beyond 0.14 wt.% could be attributed to high matrix hardening (and hence decrease in residual ductility), Fig.1b, which can result from the formation of SRO.

Observed shift in DSA temperature region to 773-873 K for N > 0.11Wt.% is justifiable, since nitrogen retards Cr-diffusivity and delays DSA due to Cr-atoms [11]. Tensile test results on the present compositions also indicated shifted in DSA regime to higher temperatures with increase in nitrogen content [22].

3.2 Strain amplitude effect

In the observed DSA temperature-regime, 773-873 K, tests have been conducted at strain amplitudes $\pm$ 0.25 to $\pm$ 1.0% at a strain rate of 3 x 10^{-3} s^{-1}. N11 and N14 steels have shown more or less similar cyclic stress strain response at all strain amplitudes and strengthening effect of nitrogen is perceived over all strain amplitudes particularly for N22 steel, as shown in Fig.3.

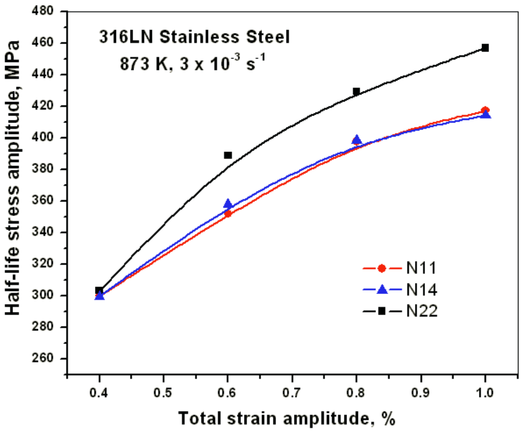


Fig.3: Influence of nitrogen on cyclic stress-strain curve at 873 K.

Cyclic stress response in the temperature range 773-873 K has been found to be similar irrespective of nitrogen content at each applied strain amplitude. From the above it is seen that, stress response in this DSA temperature range increased with increase in temperature at 0.6% strain amplitude. In contrast to above, stress response is observed to decrease with increase in temperature, irrespective of nitrogen content, for strain amplitudes beyond 0.6%, Fig.4b. As apparent from Fig.4b, effect of DSA on cyclic stress response is found to be diminishing i.e., cyclic stress response at 823 K and 873 K lies below or close to 773 K. This implies the fact that planar slip due to DSA can be overwhelmed by the increase in tendency towards cross-slip which increases with increase in strain amplitude. In such case, increase in amount of cross-slip (at high strain amplitudes) should reflect in an increase in number of crack initiation sites in contrast to those at low strain amplitudes. To observe this, replica of the surface features of the as-tested specimens was examined with optical microscopy. Interestingly, at low strain amplitudes (LSAs) secondary crack density is much lower than that those at high strain amplitudes (HSAs) which showed crack initiation events in most of the grains, Fig.5. However, most of the crack lengths are much longer in LSAs (Fig.5a) compared to those at HSAs (Fig.5b), which can be due to high fatigue lives at LSAs that allow crack coalescence to take place.

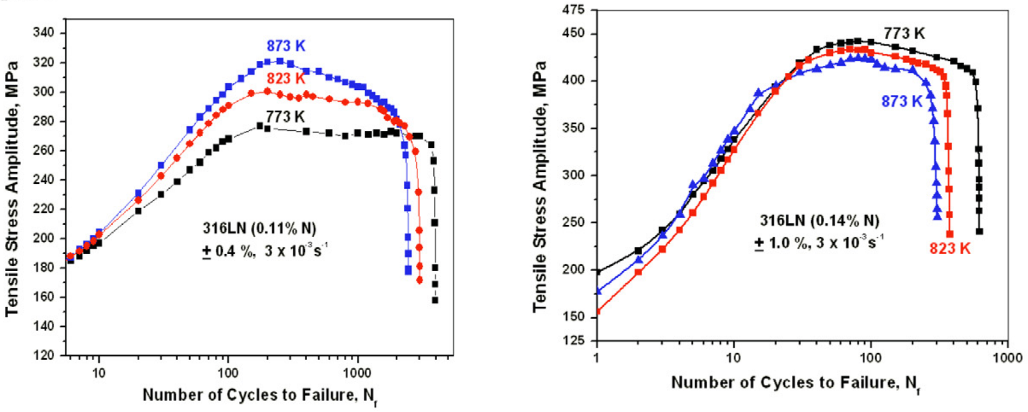


Fig.4: Effect of strain amplitude on cyclic stress response in DSA temperature region (673-873 K), at (a) ± 0.4% and (b) ± 1.0%. The above stress responses are observed to be similar irrespective of nitrogen content.

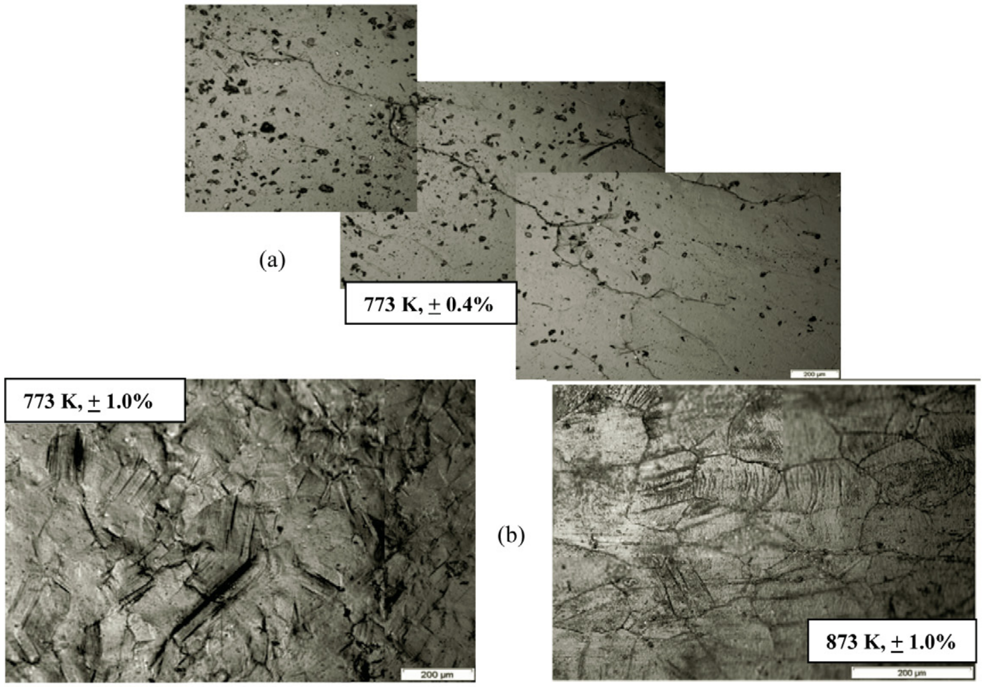


Fig.5: Change in surface crack morphologies as a function of strain amplitude in N22 steel at, (a) 773 K, ± 0.4%, (b) ± 1.0 %, at 773 K and 873 K. Acetate replica was used to capture the surface crack morphologies.

3.3 Strain rate effect

DSA being a time and temperature dependent phenomenon, gets intensified with decrease in strain rate or increase in temperature both of which accentuate the diffusion of solute atoms responsible for the occurrence of DSA. At intermediate temperatures, where the effects of time dependent processes such as creep and oxidation are found to be minimal, drastic reduction in LCF life has been observed with increasing temperature and decreasing strain rate in 304L(N) and 316L(N) stainless steels in the temperature range 773 K to 873 K [15,23].

Tests have been conducted at different strain rates (3 x 10^{-3} to 3 x 10^{-5} s^{-1}), to strengthen the evidence for DSA in the temperature range 773-873 K. The occurrence of DSA can be identified, under these conditions, through one of its manifestation i.e., increase in the period of initial hardening with decrease in strain rate (Fig.6a) or increase in temperature (Fig.1) in the DSA regime. Also, negative strain rate sensitivity of cyclic stress, i.e., increase in stress with decrease in strain rate, is evidenced in the strain rate change tests, as shown in Fig.6a at 773 K for N14 steel. Effect of DSA on reduction in fatigue life is found to be more detrimental with decrease in strain rate, Fig.6b, in comparison to that observed in temperature change tests (773-873 K, Fig.1) due to much higher matrix hardening (Fig.6a) in the former case. Such high matrix hardening could result from locking of mobile dislocations by solute atoms and /or solute-atom complexes. However, in these low strain rate regimes, copious amount of carbide precipitation has been reported in 316LN SS (with 0.78 Wt.% N), under similar test conditions [19]. It is difficult to delineate the effects of DSA and precipitation on cyclic stress response and fatigue life; the consequences of low strain rate effects are therefore attributed to the combined effects of DSA and precipitation.

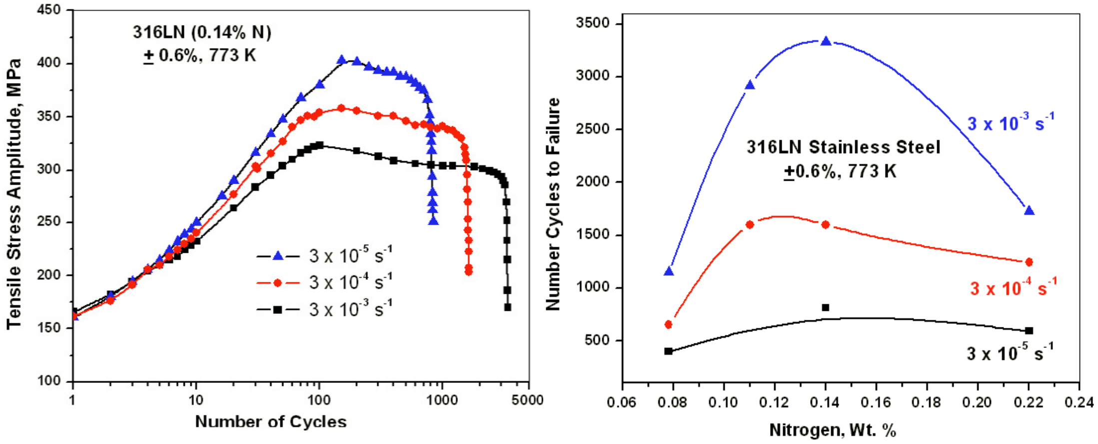


Fig.6: (a) Influence of strain rate on cyclic stress evolution at 773 K for N14 steel and (b) Effect of strain rate and nitrogen on fatigue life at 773 K.

The combined effect of strain rate, temperature and nitrogen content on fatigue life is similar to the previously observed combined effect of temperature and nitrogen on fatigue life i.e., decrease in fatigue life beyond 0.14 wt% N, Fig.7.

3.4 Life prediction in the DSA regime

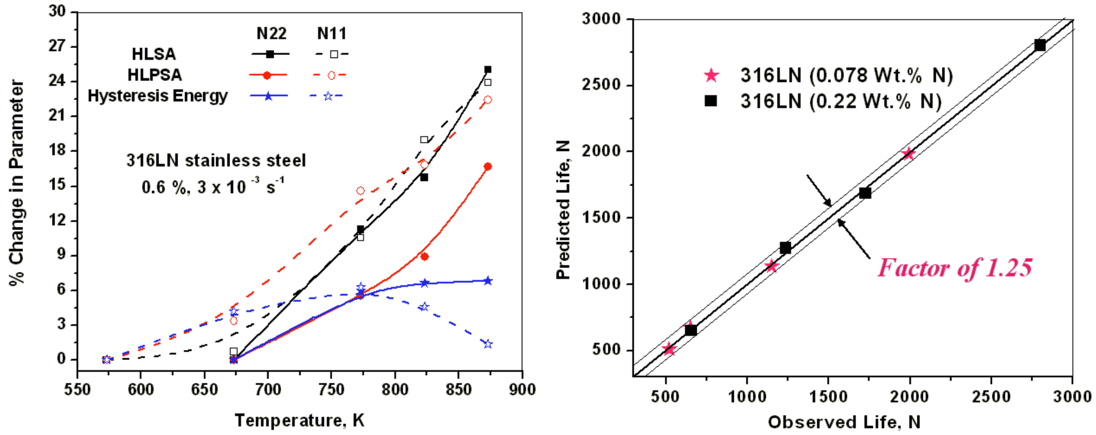
An attempt has been made to predict the life in the DSA regime (673-873 K) for the tests conducted at ± 0.6% strain amplitude with strain rate 3 x 10^{-3} s^{-1}. Firstly, to access the severity of fatigue damage induced by DSA, parameters sensitive to the occurrence of DSA were obtained from half-life hysteresis loop, such as half-life stress amplitude (HLSA), half-life plastic strain amplitude (HLPSA) and half-life hysteresis energy (HLHE). The percentage change in these parameters plotted as a function of test temperature (from 673-873 K), Fig. 8, revealed less sensitivity of hysteresis energy to the induced fatigue damage, in comparison to HLSA and HLPSA. This seems to be justifiable, since DSA leads to matrix hardening that increases HLSA and decreases HLPSA which in turn results in negligible/small change in hysteresis loop energy. Parameters HLSA (**σ**) and HLPSA (**ε_p**) are therefore used to predict the fatigue life in the DSA regime and are related to life as follows:

$$N = A.\ (\sigma/E)^{P}\ .\ (\varepsilon_p/\varepsilon_t)^{Q} \qquad (1)$$

Where A, P and Q are constants, E is Elastic modulus at the respective test temperature (determined from first quarter cycle) and **ε_t** is the total strain amplitude. Constants in the above equation are evaluated by multiple regression analysis of the experimental data. The calculated values of the constants are:

Steel	A	P	Q
N078	11	-1.39	10.3
N22	148	-0.76	6.57

With the above constants, fatigue life in the DSA region (763-873 K) has been predicted, Fig.9. As apparent from figure, predicted life is in good agreement with the observed life, within a factor of 1.25.



(a) (b)

Fig.7: (a) Percentage change in half-life stress and plastic strain amplitudes and hysteresis energy, and (b) Predicted and observed lives for N078 and N22 steels, in the DSA region (673-873 K).

4 Conclusions

1) Beneficial effect of nitrogen on fatigue life is observed to increase/saturate with increase in nitrogen content for temperatures < 673 K. For temperatures ≥ 673 K, fatigue life is found to be maximum for a nitrogen content of 0.14 wt.%.

2) Increase in nitrogen content shifted the occurrence of DSA temperature regime from 673-873 K, for nitrogen contents < 0.14 wt.%, to 773-873 K for nitrogen levels ≥ 0.14%. DSA resulted in drastic reduction in fatigue life in the temperature-strain rate domain of its occurrence; reduction being severe at low strain rates.

3) Occurrence of DSA was found to be dependent on applied strain amplitude. For total strain amplitudes beyond ± 0.6%, occurrence of DSA was minimal as evidenced by the disappearance of inverse temperature dependence and by the presence of numerous multiple crack initiation sites within the same grain suggesting the occurrence of stress-assisted cross-slip.

4) Life prediction carried out in the DSA region (673-873 K) for the tests conducted at constant strain amplitude, yielded good correlation between observed and predicted fatigue lives.

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