Available online at www.sciencedirect.com

ScienceDirect

Procedia Engineering 184 (2017) 765 – 772

Procedia Engineering

www.elsevier.com/locate/procedia

Advances in Material & Processing Technologies Conference

Dynamic Strain Ageing Behaviour of Modified 9Cr-1Mo Steel under Monotonic and Cyclic Loading

N. C. Santhi Srinivas*, Preeti Verma and Vakil Singh

Department of Metallurgical Engineering, Indian Institute of Technology (Banaras Hindu University), 221005, Varanasi, India

Abstract

Dynamic strain ageing (DSA) behavior of modified 9Cr-1Mo steel under uniaxial tensile and constant strain cyclic loading is presented in this paper. Tensile tests are carried out at different strain rates from 10^{-3} to 10^{-5} s^{-1} in the temperature range from room temperature to 600 °C. DSA has been established in the temperature range between 250 and 400°C in terms of peak in tensile strength, minima in ductility, serrated plastic flow and negative strain rate sensitivity. In the region of DSA dislocation substructure revealed high density of dislocations and features like kinks and bowing of dislocations are observed. Low cycle fatigue behavior was studied at 300°C, over a wide range of strain amplitudes at different strain rates. An inverse effect of strain rate was observed on cyclic stress response and fatigue life at 300 °C due to DSA.

Peer review under responsibility of the organizing committee of the Advances in Materials & Processing Technologies Conference

Keywords: Modified 9Cr-1Mo steel; Dynamic Strain Ageing; Serrated Flow; Low Cycle Fatigue.

1. Introduction

In last decades, the ferritic/martensitic 9-12 % Cr steels have been most widespread for power plants applications [1-3]. Among all the ferritic/martensitic steels, modified 9Cr–1Mo steel is very important because of its higher thermal conductivity, lower thermal expansion coefficient, higher tensile and creep strength, better weldability, higher microstructure stability and resistance to stress corrosion cracking in water–steam systems [4-6]. During service, steam generator components are exposed to high temperatures. The temperature gradients in conjunction

Corresponding author.
E-mail address: ncssrinivas.met@iitbhu.ac.in

Peer review under responsibility of the organizing committee of the Advances in Materials & Processing Technologies Conference
doi:10.1016/j.proeng.2017.04.155

with operating loads can cause plastic strains to develop low cycle fatigue (LCF) in the component. Therefore, LCF represents a vital damage phenomenon at many occasions. Fatigue failure of materials at elevated temperatures is influenced by various time and temperature dependent damage processes such as creep, phase transformations and dynamic strain ageing (DSA) in the temperature range of their services [7-8]. Different manifestations of DSA reported by several investigators include increased ultimate tensile strength, negative strain–rate sensitivity, reduced ductility, serrations in the stress–strain plots, higher dislocation density, and increased work–hardening index (n) [7]. In certain applications, the strengthening effects of DSA can be used to advantage [9]. DSA has also been found to be deleterious to tensile ductility, LCF life, and creep resistance of structural components [7-8]. During tensile testing of conventional and modified 9Cr-1Mo steel in normalized and tempered condition, occurrence of DSA was reported [10-15]. In the modified 9Cr-1Mo steel, serrations appeared from the beginning of the plastic deformation and disappeared before the ultimate tensile strength and serrations were established to be associated with interaction of dislocations with interstitial atoms. However, in the conventional 9Cr-1Mo steel, serrations were reported in the stress-strain curve after a critical deformation and to be associated with substitutional elements [11]. The effect of microstructure on the critical strain for the onset of serrated flow in modified 9Cr–1Mo steel has been studied by Chandravathi et al. [10] by soaking the specimens at different temperatures from 800–1350°C and tensile testing in 300°C. The critical plastic strain to onset of serrated flow was found to increase with hardness of the steel which is influenced by the microstructures [10].

Low cycle fatigue behavior of modified 9Cr-1Mo steel has been studied by many researchers at different temperatures and strain rates [16-22], however, very little attention has been paid on LCF in temperature regime of DSA. Decrease in fatigue life due to DSA has been reported by several investigators in ferritic–martensitic steels such as JLF–1[23], AISI 430F [24] and austenitic stainless steel [8,25,26]. Influence of temperature and strain rate has been studied earlier under cyclic loading in modified 9Cr–1Mo steel at strain amplitude of ±0.60% and DSA was observed in the temperature range 300 to 400°C [22].

The present study deals with detailed investigation of tensile properties of the modified 9Cr-1Mo steel in normalized and tempered condition over a wide range of temperature from room temperature to 600°C, at different strain rates (10^{-3} s^{-1} to 10^{-5} s^{-1}) and its deformation behavior. Influence of strain rate on the low cycle fatigue behavior is also studied to assess the effect of DSA in this steel.

2. Material and Methods

The material of present study, the modified 9Cr-1Mo steel, was supplied by IGCAR, KALPAKKAM, India, in normalized (1060 °C/1h) and tempered (780 °C/1h) condition. The chemical composition of the steel is given in Table 1.

Table 1. The chemical composition of the modified 9Cr-1Mo steel (wt. %).

Elements	C	Si	Mn	P	S	Cr	Mo	Ni	Al	Nb	N	V	Fe
wt. %	0.1	0.26	0.41	0.018	0.002	9.27	0.95	0.33	0.013	0.074	0.044	0.21	Balance

Cylindrical tensile specimens were machined from the as received material with gage diameter of 4.5 mm and gage length of 15 mm. Tensile tests were conducted from room temperature (RT) to 600 °C for different strain rates from 10^{-3} s^{-1} to 10^{-5} s^{-1} using 100 kN screw–driven Instron™ Universal Testing Machine (Model: 4206). Low cycle fatigue specimens of gage diameter 5.5 mm and gage length 12 mm were prepared from the blanks. Isothermal low cycle fatigue tests were carried out under fully reversed strain control loading using triangular waveform at different strain amplitudes from ±0.25% to ±0.50% at strain rates of 10^{-2} s^{-1} & 10^{-3} s^{-1} at elevated temperature of 300 °C. All the tests were performed in air. The test temperatures for the both types of tests, tensile as well as LCF, were controlled by a resistance heating furnace and spot-welded thermocouple on the sample. The experimental procedure at elevated temperature consists of heating the specimen from RT to the test temperature and exposing the sample at that temperature for 30 minutes to homogenize the temperature in the specimen before starting the test. Deformation behavior of the tested samples was studied by transmission electron microscope (Model: Technai-G^2-20). TEM foils were prepared from thin slices sectioned at a distance of 1 mm from the fracture end, mechanically polishing up to 50 µm and electro-polishing in the electrolyte containing 6% perchloric acid, 34% n-butanol and 60% methanol cooled to -40 °C, at 20 V.

3. Results and Discussion

3.1. Tensile Behavior

Engineering stress-strain curves corresponding to different strain rates at different temperatures; RT, 300, and 600 °C are shown in Fig.1. It is quite obvious from the Fig. 1(a) and 1(c) that both yield and ultimate tensile strength decrease with decrease in strain rate from 10^{-3} s^{-1} to 10^{-5} s^{-1} at RT and 600 °C. On the other hand, at the intermediate temperature of 300 °C (Fig. 1(b)), there is inverse effect of the strain rate. It may also be seen that ultimate tensile strength increased with decrease in strain rate. Serrated flow in the stress-strain curve corresponding to strain rate of 10^{-4} s^{-1} at 300 °C may be seen from the magnified view of the encircled portion. However, at the other strain rates smooth plastic flow was observed. The occurrence of serrations in the stress-strain curve was found to be in line with the earlier findings [11].

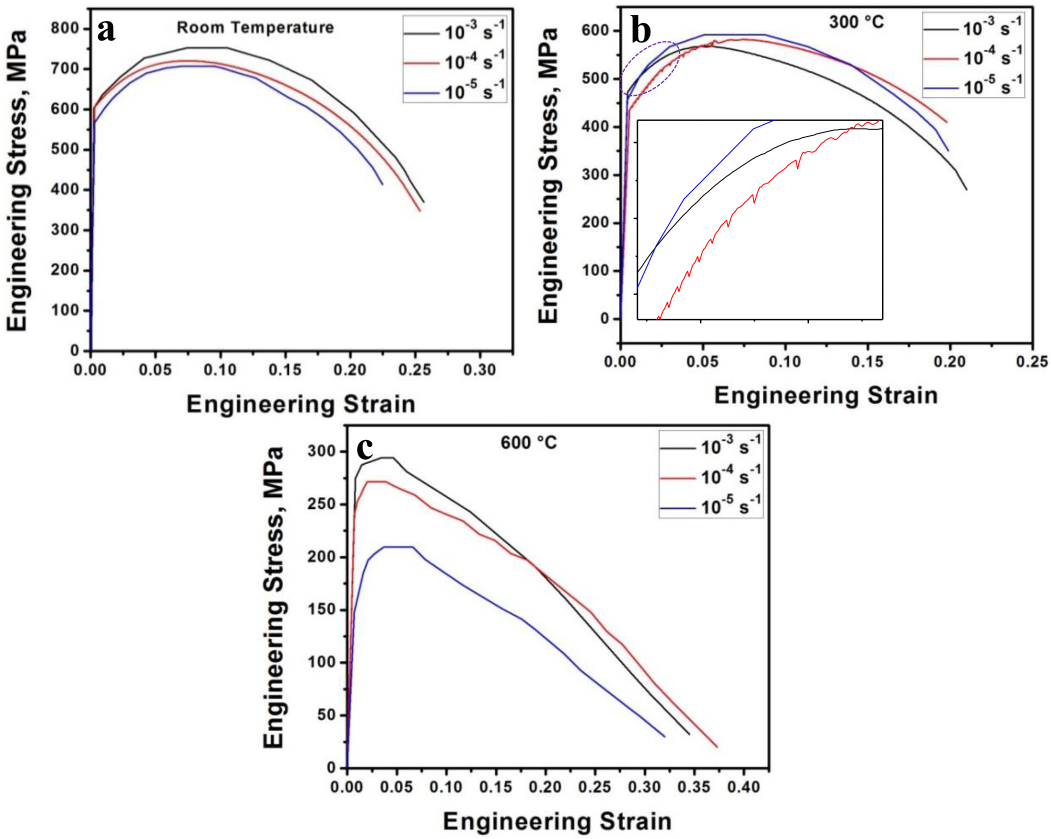

Fig. 1. Engineering stress-strain curves for different strain rates at different temperature (a) RT, (b) 300 °C, and (c) 600 °C.

Variation of true ultimate tensile strength with strain rate at different temperatures is shown in Fig. 2(a). It may be noted from the figure that at RT and 600 °C, yield and tensile strengths were decreased with decrease in strain rate and displayed positive slope. However, UTS increased with decrease in strain rate between 250-400 °C and negative values of strain rate sensitivity are obtained as given in the Table 2.

Figure 2(b) shows variation of true ultimate tensile strength and percent total engineering elongation with temperature at different strain rates. It is quite obvious from the plot that there is rapid drop in strength from RT to 200 °C and from 400 to 600 °C at all the strain rates. A pronounced peak was observed in the strength at strain rate of 10^{-4} s^{-1} at the intermediate temperature. Relatively slight increase in strength was also observed in the temperature range from 250 to 400 °C, at the strain rates of 10^{-3} s^{-1} and 10^{-5} s^{-1}. Total elongation was found to be decreased with increase in temperature from RT to 350 °C and subsequently increased up to 600 °C. It may be seen that valley in the total elongation was observed in the intermediate temperature range from 200 to 400 °C. It is important to mention that the minimum elongation temperature was found to shift towards the lower temperature with decrease in strain rate. Anomalous variation in the tensile properties with respect to the temperature and strain rate confirms the occurrence of DSA in this modified 9Cr-1Mo steel in the intermediate temperature range from 250-400 °C [7] and is also in good agreement with the earlier studies on 9Cr-1Mo steel [11-12].

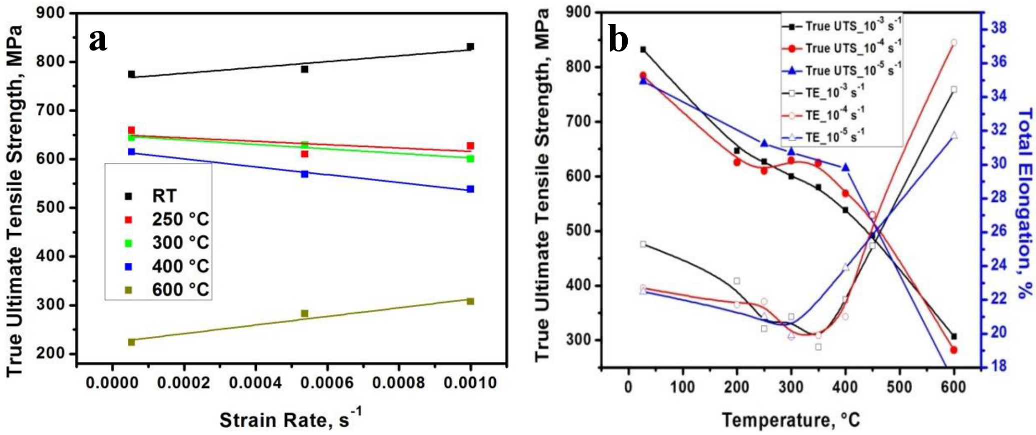

Fig. 2. **(a)** Showing the variation of ultimate tensile strength with strain rate, **(b)** Variation of true ultimate tensile strength and total elongation in % with temperature at different strain rates.

Table 2. Strain rate sensitivity at different temperatures at strain rate of 10^{-4} s^{-1}.

Temperature (°C)	Strain Rate Sensitivity
27	0.01921
250	-0.0216
300	-0.02037
400	-0.04232
600	0.10651

Effect of occurrence of DSA on the dislocation substructure in modified 9Cr-1Mo steel has been studied using TEM. For the comparison, two samples were investigated which were tested at 200 °C (non-DSA region) and 350 °C (maximum DSA region) at strain rate of 10^{-4} s^{-1}. Bright field TEM micrograph of the as received modified 9Cr-1Mo steel in normalized and tempered condition is shown in Fig. 3(a). Tempered martensitic lath structure inside the prior austenite grain boundaries can be seen. Distribution of second phase particles along the prior austenite grain boundaries, lath boundaries and within the laths may also be seen. These second phase particles were identified as $M_{23}C_6$, vanadium and niobium carbides [21]. This steel exhibits high dislocation density in the normalized and tempered condition. Bright field TEM micrograph of the sample tested at temperature of 350 °C and strain rate of 10^{-4} s^{-1} which has the maximum DSA region, showed high dislocation density in comparison to the sample tested at 200 °C (Fig. 3(b)). Typical features of DSA may also be seen from the micrograph taken at higher magnification such as dislocation-dislocation interaction and bowing of dislocation (Fig. 3(c)).

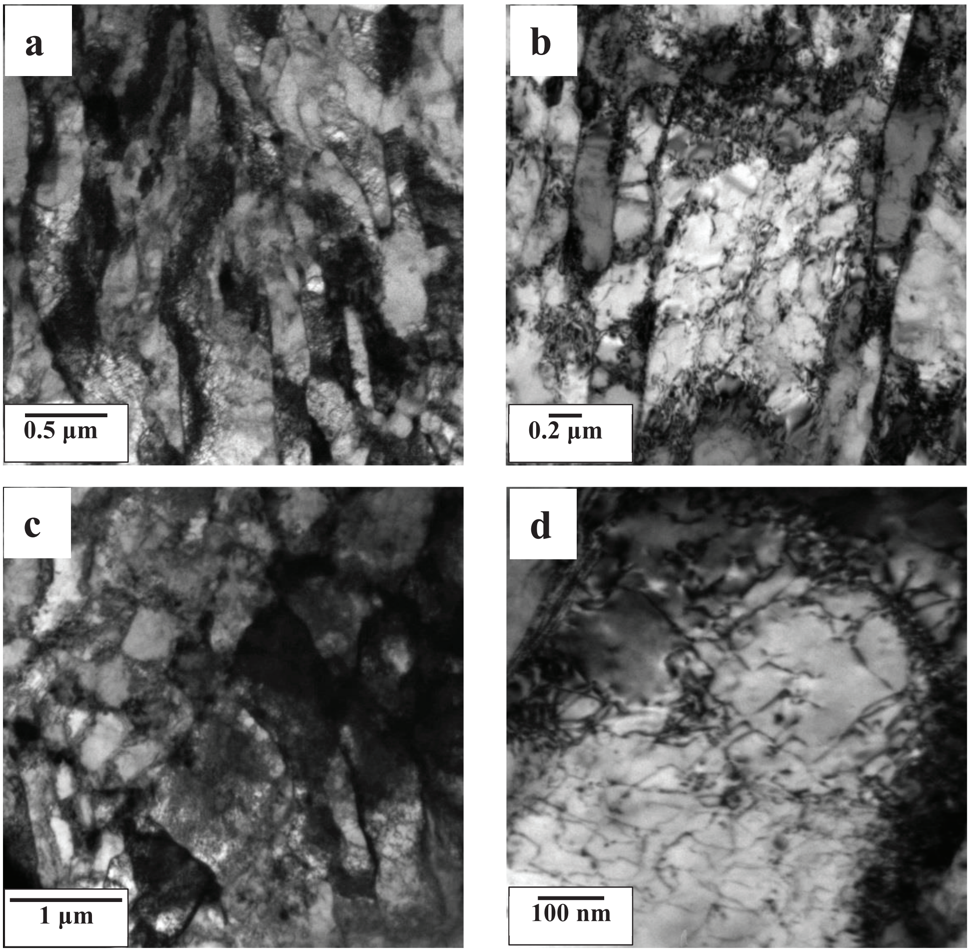

Fig. 3. (a) Bright field TEM micrographs of the modified 9Cr-1Mo steel in normalized and tempered condition, (b) tensile tested at 200 °C, (c) and (d) tensile tested at 350 °C.

3.2. Low Cycle Fatigue Behavior

Variation of low cycle fatigue life of modified 9Cr-1Mo steel with plastic strain amplitude at 300 °C is shown in Fig. 4(a). It is obvious from the plot that there is a linear relationship between fatigue life and the plastic strain amplitude at both the strain rates and obeyed the Coffin-Manson relationship. It can also be seen from the figure that fatigue life is reduced at the lower strain rate of 10^{-3} s^{-1} in comparison to that of 10^{-2} s^{-1}. This may be more appreciated from Fig. 4(b). In general, fatigue life increases with decrease in strain rate. However, in the present investigation, fatigue life was decreased at lower strain rate of 10^{-3} s^{-1} in comparison to that of higher strain rate of 10^{-2} s^{-1}. Thus, inverse dependency of strain rate on fatigue life may be associated with dynamic strain ageing at this temperature. It further confirms the inverse strain rate sensitivity observed in the temperature range between 250 and 400 °C. It has been reported in literature that DSA under cyclic deformation causes reduction in fatigue life [25-26].

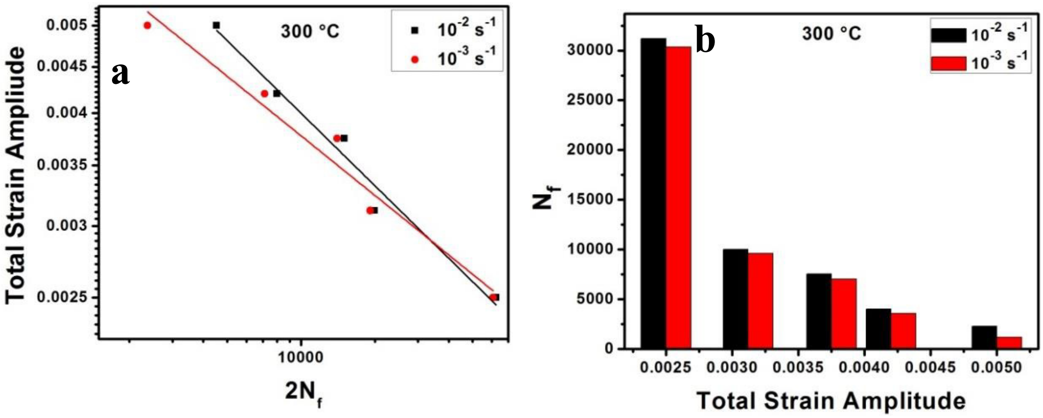

Fig. 4. (a) Variation of number of reversals to failure with total strain amplitude at both strain rates of 10^{-2} s^{-1} and 10^{-3} s^{-1} (b) Bar graph showing the fatigue life for different strain amplitudes at both strain rates.

Cyclic stress response of modified 9Cr-1Mo steel tested at 300 °C at total strain amplitude of ±0.50% at both strain rates is shown in Fig. 5. It may be seen from the figure that there is tendency of mild hardening from first cycle to 10th cycle followed by continuous softening till failure at the lower strain rate of 10^{-3} s^{-1}. However, at high strain rate of 10^{-2} s^{-1}, decrease in stress amplitude from first cycle to second followed by stabilization and subsequent continuous softening till failure. The high rate of hardening at lower strain rate of 10^{-3} s^{-1} in comparison to that at 10^{-2} s^{-1} may be associated with the DSA. Extent of hardening is higher at the lower strain rate of 10^{-3} s^{-1} in comparison to that of 10^{-2} s^{-1}.

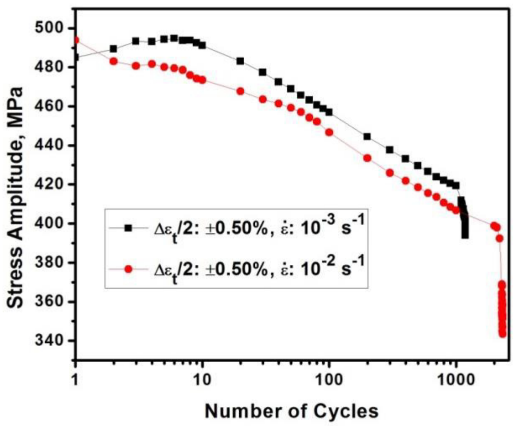

Fig. 5. Cyclic stress response of modified 9Cr-1Mo steel tested at 300 °C at total strain amplitude of ±0.50% at strain rates of 10^{-2} s^{-1} & 10^{-3} s^{-1}.

Bright field TEM micrographs of the fatigue tested samples at 300 °C, at the strain amplitude of ±0.50% at both the strain rates, are shown in Fig. 6. Formation of cell structure may be seen in both the conditions. Thus, the observed cyclic softening in the modified 9Cr-1Mo steel may be attributed to formation of dislocation cell structure. Bowing of dislocation may also be seen in the sample tested at strain rate of 10^{-3} s^{-1}. Thus, inverse dependency of strain rate on low cycle fatigue behavior at 300 °C may be understood in terms of pinning and unpinning of dislocations, which reflects the DSA behavior.

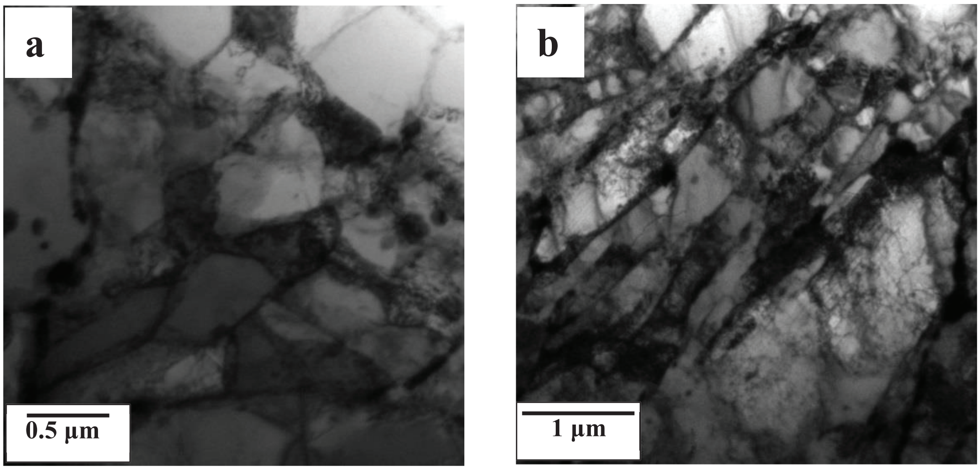

Fig. 6. Bright field TEM micrographs of the fatigue tested samples at 300 ºC at stain amplitude of ±0.50% at different strain rates: (a) 10^{-2} s^{-1} and (b) 10^{-3} s^{-1}.

4. Conclusions

- The modified 9Cr-1Mo steel was found to exhibit dynamic strain ageing in the temperature range from 225 to 400 °C.
- DSA was established on the basis of serrations in the stress-strain curves, plateau/peak in strength and minima in ductility.
- The modified 9Cr-1Mo steel was found to exhibit cyclic softening irrespective of strain amplitude and strain rate at 300 °C and was associated with the formation of cell structure.
- Decrease in fatigue life and increase in stress amplitude at the lower strain rate in comparison to the strain rate of 10^{-2} s^{-1} may be associated with dynamic strain ageing.

References

[1] R. Klueh, D.R. Harries, High chromium ferritic and martensitic steels for nuclear applications, ASTM, Philaedelphia, 3 (2001) 103-112.
[2] R.Klueh, Chromium-Molybdenum steels for fusion reactor first walls-A review," Nucl. Eng. Des. 72(1982)329–344.
[3] V.K. Sikka, C.T.C Ward, K.Thomas, Ferritic steels for high temperature applications, ASM Int., Metals Park, (1983)65–84.
[4] R. Nandakumar, S. Athmalingam, V. Balasubramaniyan, S.C. Chetal, Steam generators for future fast breeder reactors, Energy Procedia, 7(2011)351–358.
[5] Raj B., Indira Gandhi Centre for Atomic Research, Annual Technical Report, (2010)1–248.
[6] S.L.Mannan, S.C. Chetal, B. Raj, S.B. Bhoje, Selection of materials for prototype fast breeder reactor. T. Indian Metals, 56 (2003)155–178.
[7] S. Mannan, Role of dynamic strain ageing in low cycle fatigue, Bull. Mater. Sci. 16(1993)561–582.
[8] K.B.S. Rao, M. Valsan, R. Sandhya, S.L. Mannan, P. Rodriguez, Dynamic strain ageing effects in low cycle fatigue, High Temp. Mater. Processes, 7(1986)171–178.
[9] J.D. Baird , The effects of strain ageing due to interstitial solutes on the mechanical properties of metals, Metall. Review 16 (1971) 1-18.
[10] K.S. Chandravathi, K. Laha, P. Parameswaran, M.D. Mathew, Effect of microstructure on the critical strain to onset of serrated flow in modified 9Cr–1Mo steel, Int. J. Pres. Ves. Pip. 89(2012)162–169.
[11] R. Kishore, R.N. Singh, T.K. Sinha, B.P. Kashyap, Effect of dynamic strain ageing on the tensile properties of a modified 9Cr–1Mo steel, J. Mater. Sci. 32(1997)437–442
[12] C. Keller, M.M.Margulies, Z. Hadjem-Hamouche, and Guillot I., Influence of the temperature on the tensile behaviour of a modified 9Cr–1Mo Steel," Mater. Sci. Eng. A. 527(2010) 6758–6764.
[13] R. Kishore, R. Singh, T. Sinha, B.P. Kashyap, Serrated flow in a modified 9Cr–1Mo steel, Scripta Metall. et Mater. 32(1995) 1297–1300.

[14] A.K. Roy, P. Kumar, D. Maitra, Dynamic strain ageing of p91 grade steels of varied silicon content," Mater. Sci. Eng. A 499 (2009) 379–386.
[15] R. Kishore T.K. Sinha, Analysis of the stress–strain curves of a modified 9Cr–1Mo steel by the Voce equation," Metall. Mater. Trans. A 27 (1996) 3340–3343.
[16] K.Guguloth, S.Sivaprasad, D. Chakrabarti, S. Tarafder, Low-cyclic fatigue behavior of modified 9Cr-1Mo steel at elevated temperature, Mater. Sci. Eng. A 604 (2014) 196–206.
[17] S. Kim, J.R. Weertman, Investigation of microstructural changes in a ferritic steel caused by high temperature fatigue, Metall. Trans. 19A (1988) 999–1007.
[18] A. Nagesha, M. Valsan, R. Kannan, K.B.S. Rao, S.L. Mannan, Influence of temperature on the low cycle fatigue behaviour of a modified 9Cr–1Mo ferritic steel, Int. J. Fatigue 24 (2002) 1285–1293.
[19] V. Shankar, M. Valsan, K.B.S. Rao, R. Kannan, S.L. Mannan, S.D. Pathak, Low cycle fatigue behavior and microstructural evolution of modified 9Cr-1Mo ferritic steel, Mater. Sci. Eng. A 437 (2006) 413–422.
[20] P. Verma, N.C. Santhi Srinivas, V. Singh, Low cycle fatigue behaviour of modified 9Cr–1Mo steel at 600 °C, T. Indian I. Metals 69 (2016) 331–335.
[21] P. Verma, N.C.S. Srinivas, S.R. Singh, V. Singh, Low cycle fatigue behavior of modified 9Cr-1Mo steel at room temperature, Mater. Sci. Eng. A 652 (2016) 30–41.
[22] K. Mariappan, V. Shankar, R. Sandhya, G.V. Prasad Reddy, M.D. Mathew, Dynamic strain aging behavior of modified 9Cr-1Mo and reduced activation ferritic martensitic steels under low cycle fatigue, J. Nucl. Mater. 435 (2013) 207–213.
[23] H. Li, A. Nishimura, T. Nagasaka, T. Muroga, Stress-strain behavior on tensile and low cycle fatigue tests of JLF-1 steel at elevated temperature in vacuum, Fusion Eng. Des. 81 (2006) 2907–2912.
[24] M. Avalos, A.F. Armas, Dynamic strain aging effects on low-cycle fatigue of AISI 430F, Mater. Sci. Eng. A 514 (2009) 1–7.
[25] K.Tsuzaki, T.Hori, T.Maki, I.Tamura, Dynamic strain aging during fatigue deformation in type 304 austenitic stainless steel, Mater. Sci. Eng. A 61(1983) 247–260.
[26] A.Armas, O.Bettin, I.Alvarez-Armas, Strain aging effects on the cyclic behavior of austenitic stainless steels, J. Nucl. Mater. 155-157 (1988) 644–649.