Effect of sodium environment on the low cycle fatigue properties of modified 9Cr–1Mo ferritic martensitic steel

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ABSTRACT

Modified 9Cr–1Mo ferritic steel is the material of current interest for the steam generator components of liquid metal cooled fast breeder reactors (LMFBRs). The steam generator has been designed to operate for 30–40 years. It is important to accurately determine the life of the components in the actual environment in order to consider the extension of life beyond the design life. With this objective in view, a programme has been initiated at our laboratory to evaluate the effects of flowing sodium on the LCF behaviour of modified 9Cr–1Mo steel. LCF tests conducted in flowing sodium environment at 823 K and 873 K exhibited cyclic softening behaviour both in air and sodium environments. The fatigue lives are significantly improved in sodium environment when compared to the data obtained in air environment under identical testing conditions. The lack of oxidation in sodium environment is considered to be responsible for the delayed crack initiation and consequent increase in fatigue life. Comparison of experimental lifetimes with RCC-MR design code predictions indicated that the design curve based on air tests is too conservative.

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1. Introduction

Modified 9Cr–1Mo steel is used extensively as structural material for steam generator components of liquid metal cooled fast breeder reactor and fossil fired power plants. The selection of this material is primarily based on a good combination of mechanical properties, high thermal conductivity, low thermal expansion coefficient and good resistance to stress corrosion cracking in water–steam environment systems compared to austenitic stainless steels. In comparison with plain 9Cr–1Mo steel, the modified 9Cr–1Mo alloy exhibits better monotonic tensile and creep strengths at temperatures higher than 773 K. This alloy also exhibits good weldability and microstructural stability over very long periods of exposure to high temperature service conditions.

The components of the steam generators are often subjected to repeated thermal stresses as a result of temperature gradients that occur because of heating and cooling during start-ups and shut-downs or during temperature transients. This leads to low cycle fatigue damage in the components. Furthermore, since the components fabricated from modified 9Cr–1Mo steel are to be used for 30–40 years in service, it is important that this alloy exhibits good microstructural stability over very long periods of exposure to high temperature service conditions. The LCF behaviour of this steel has been reported earlier under normalized and tempered [1–8] and thermally aged conditions [9–11] in air environment. Prolonged

ageing of the alloy at elevated temperatures prior to testing was found to reduce the LCF and creep–fatigue interaction lifetimes [9–11]. Ageing resulted in the formation of Laves phase with associated reduction in the toughness and LCF lifetimes of the alloy [5,7].

Liquid sodium has been chosen as the heat transfer medium in LMFBRs, because of its high thermal conductivity and heat capacity, and remain as liquid over a wide range of temperatures. The use of liquid sodium requires the assessment of the compatibility with structural materials with sodium. At present, the design of the components is based on the experimental data from tests performed in air. Environment can have a significant influence on the elevated temperature mechanical properties [12–15] and hence, it is essential to assess the environmental effects on mechanical properties of various components of the system. Performing mechanical tests in sodium, particularly under dynamic sodium environment to simulate the reactor conditions, is a very difficult task and also expensive. Only limited data are available concerning the effects of flowing sodium on mechanical properties of ferritic–martensitic steels.

Andrews and Kirschller [16] have reported that the fatigue endurance of 2.25Cr–1Mo steel in the low oxygen environments like helium and in sodium containing 30 ppm and 300 ppm oxygen, respectively, were 5–10 times higher compared to that in air. A threefold increase of fatigue endurance was reported under vacuum on a Fe–Cr–1/2Mo steel at 773 K [17]. Wood et al. [18] have reported a marginal increase in the fatigue life of 9Cr steel under impure helium environment at 798 K. All these results indicate

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that the low cycle fatigue endurance of ferritic–martensitic steels under non-oxidizing environment is better than in air. This paper deals with the low cycle fatigue properties of a modified 9Cr–1Mo steel under the flowing sodium environment.

2. Experimental details

The chemical composition of the modified 9Cr–1Mo employed in this study is given in Table 1. This steel was procured from Rourkela Steel Plant, Rourkela, India in the form of 8000 × 2000 × 30 mm plates in the rolled condition. Bars of 110 mm length and 22 mm diameter machined from the mill annealed plates were subjected to a normalizing treatment at 1313 K for one hour followed by a tempering treatment at 1033 K for one hour. This heat treatment yielded a tempered martensitic structure, made of lath martensite, $M_{23}C_6$ carbides located on prior austenite grain and lath boundaries, and a very fine secondary carbides of MX type in the lath interiors (Fig. 1). Cylindrical specimen with a gauge diameter of 9 mm and a gauge length of 21 mm (Fig. 2) were used in this investigation.

A chamber has been designed and developed in-house for conducting LCF tests in dynamic sodium environment with special emphasis on safety for avoiding leaks and accidental contact with air environment and the details are given in Fig. 3. The specimen is enclosed within a miniature primary bellow surrounding which, a secondary bellow is provided for safety. Two flanges are provided from the upper and lower portions of the specimen gauge length from which an arrangement was made to house the linear variable differential transducer for strain measurement.

Low cycle fatigue tests on modified 9Cr–1Mo steel were performed following ASTM E606 standard under fully reversed total axial strain control mode and employing a triangular waveform. Tests were conducted at 823 K and 873 K at a constant strain rate of 3×10^{-3} s^{-1} with strain amplitudes varying from ±0.4% to ±1.0%. The fatigue crack initiation and propagation modes under different testing conditions were studied using optical and scanning electron microscopy. Samples for the optical metallography were etched using Vilella's reagent (1 g of picric acid + 5 ml conc.
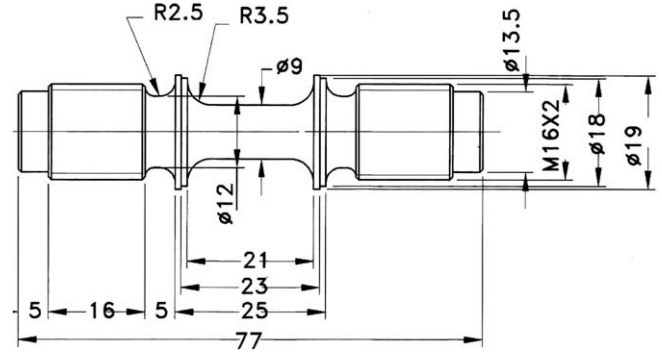

Fig. 2. Dimension of specimen used for in-sodium LCF test.

HCl + 100 ml of ethyl alcohol) and examined under an optical microscope. The fractured surfaces of the fatigue tested samples were examined by a PSEM 501 scanning electron microscope to determine the crack initiation and propagation modes. Sodium temperature variation was smaller than ±2 K and sodium velocity was maintained at 2.5 m/s in the test chamber. The oxygen level in the sodium loop was maintained at <2 ppm.

3. Results and discussion

3.1. Cyclic stress response

A representative comparison of the cyclic stress response under air and flowing sodium environments is given in Fig. 4. The alloy exhibited a continuous softening before the final load drop that occurred due to the propagation of fatigue macrocracks. The cyclic stress response increased with the increase in strain amplitude. Cyclic softening has been reported by several others in ferritic–martensitic steel [1,4,5,19]. The mechanisms that have been suggested to explain the softening in Cr–Mo steels based on extensive substructural investigations include [10,19,20]: (i) annihilation of dislocations introduced during martensitic transformation,

Table 1
Chemical composition of indigenous modified 9Cr–1Mo steel (in wt%).

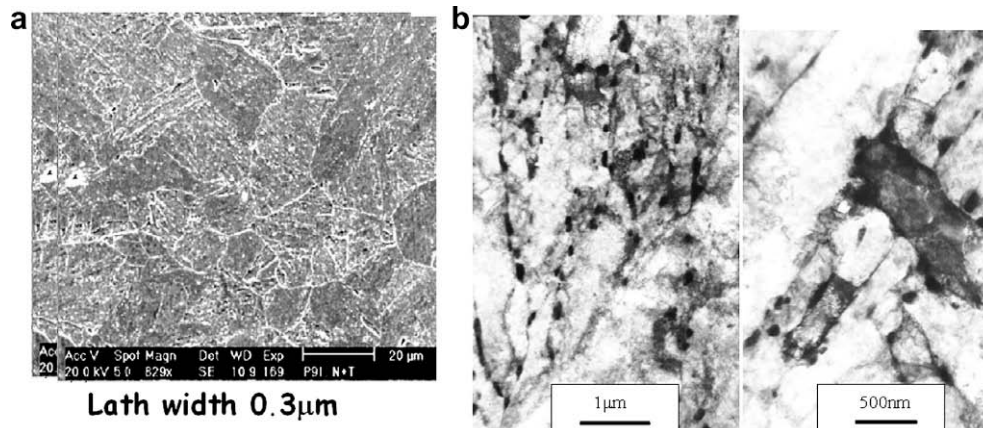| C	Cr	Mo	Mn	Nb	V	Ni	Si	N	Al	S	P
0.11	9.28	0.98	0.38	0.099	0.25	0.13	0.465	0.068	0.21	0.007	0.020

Fig. 1. (a) Optical microstructure of modified 9Cr–1Mo steel depicting tempered martensitic structure and (b) TEM micrograph showing $M_{23}C_6$ precipitates along the prior austenite grain boundaries and fine MC type precipitates in the lath interiors.
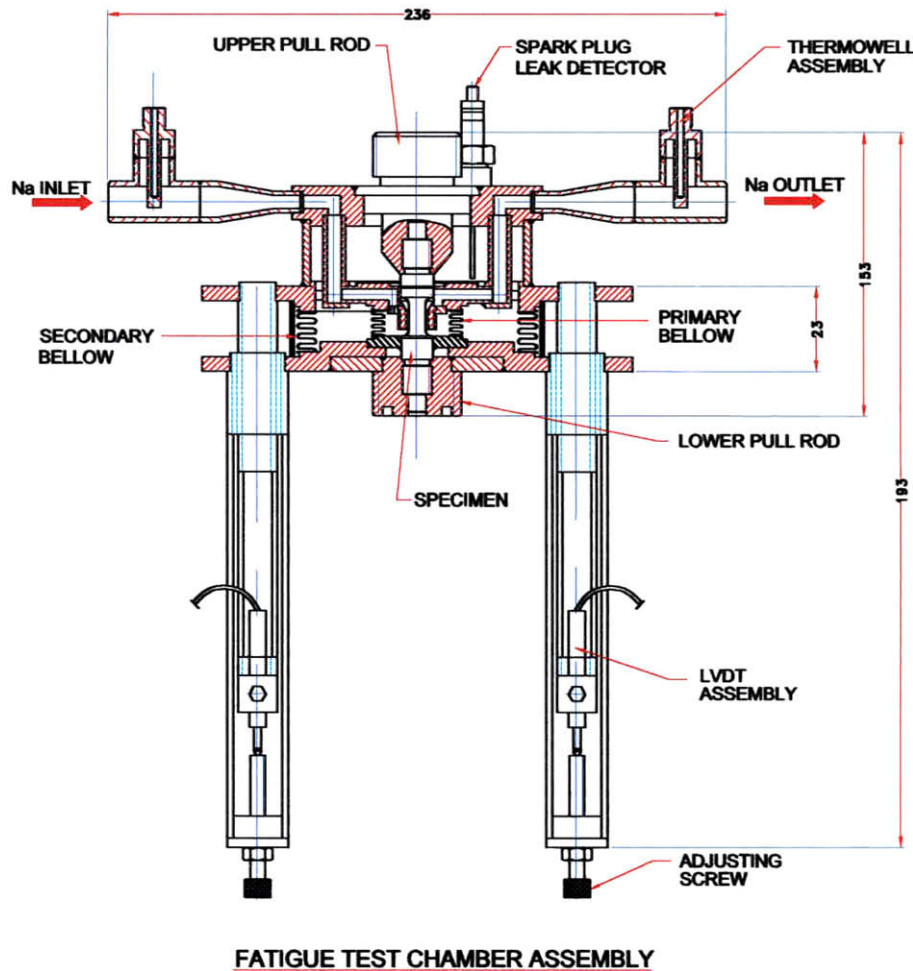
Fig. 3. Details of fatigue test chamber.

(ii) change from the original lath structure to cells or equi-axed subgrains, (iii) degradation of strength due to coarsening of precipitates, (iv) replacement of Mo–C pairs by Mo–C–Mo clusters with an associated loss in interaction solid solution hardening and (v) re-solution by which the metastable strengthening precipitates completely dissolve in the matrix after being cut by dislocations
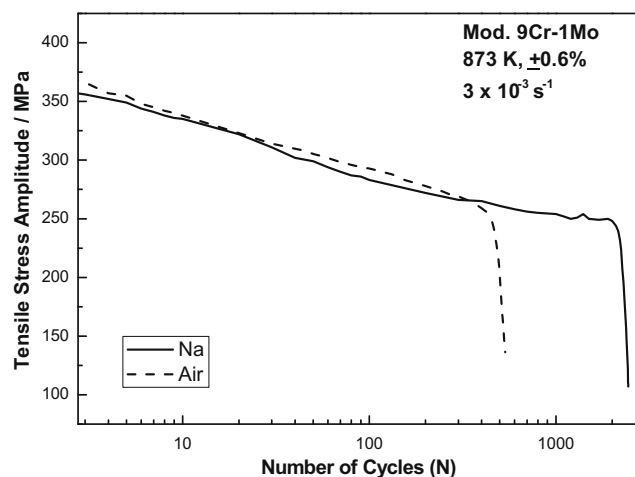

Fig. 4. Influence of environment on the cyclic stress response of modified 9Cr–1Mo steel.

to a size smaller than the critical size for particle nucleation. Following the literature, the short LCF lifetimes at these temperatures do not affect strongly the precipitates and therefore cyclic softening is not due to precipitate evolution. An earlier study in this material in our laboratory [19] has shown that during high temperature LCF tests in air, the initial lath structure comprising of high dislocation density at lath boundaries is transformed into a low energy configuration of cells leading to cyclic softening. The cyclic softening of the alloy in air and dynamic sodium environment (Fig. 4) is very much similar. Similar behaviour was observed by Kim and Weertman [10] under vacuum environment. The cyclic softening in sodium environment continued over a large number of cycles.

3.2. Fatigue life and fracture behaviour

Table 2 summarizes the influence of flowing sodium on the LCF life of modified 9Cr–1Mo steel at 823 K (Fig. 5). Fig. 6 depicts the influence of sodium on LCF life at 873 K. The results clearly indicate that there is a remarkable increase in the fatigue lifetimes in sodium.

The effects of environment on the mechanical properties can be divided into two classes: the influence of environment itself and the effects of microstructural and compositional changes that occur in the material during the long-term exposure to the environment. Liquid metals can influence the surface-active properties of the material through liquid metal embrittlement (LME), oxidation,

Table 2
LCF properties of modified 9Cr–1Mo steel in sodium and air environment at 823 K.

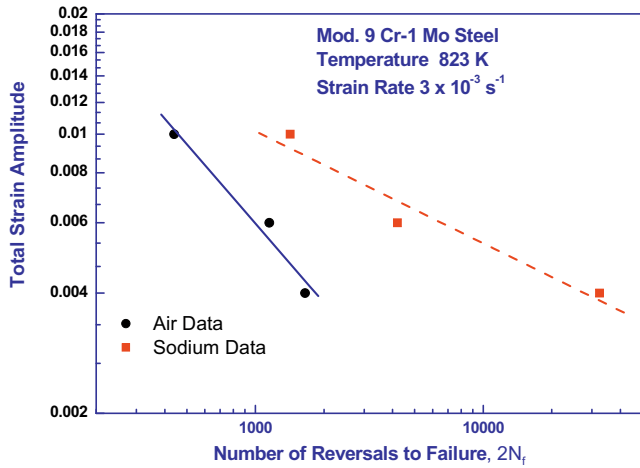| Total strain amplitude $(\Delta\varepsilon_t/2)$ %	Half-life stress (MPa) in sodium	Number of cycles to failure in sodium (N_f)	Number of cycles to failure in air (N_f)	Factor of life increase in sodium
0.4	247	16200	826	19.6
0.6	261	2100	575	3.65
1.0	345	710	220	3.2

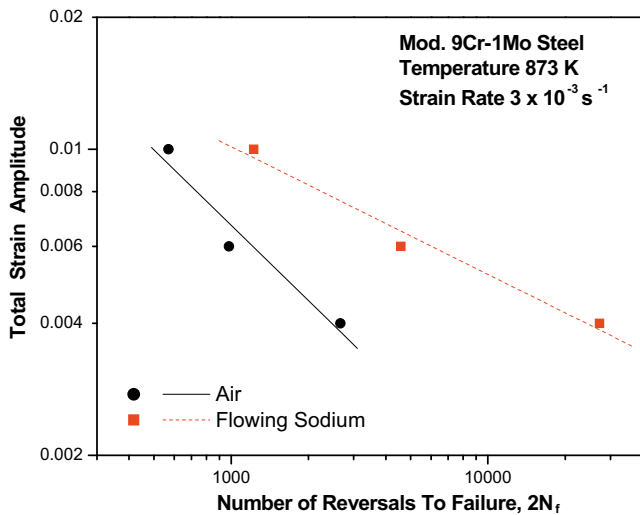**Fig. 5.** Strain–life plot at 823 K.

Fig. 6. Strain–life plot at 873 K.

nitridation, or carburization–decarburization phenomena, all of which can alter near-surface deformation behaviour and thus affect fatigue crack initiation and propagation. In modified 9Cr–1Mo steel LME occurs below 723 K and above that temperature the occurrence of LME is only in the post UTS region of stress strain curve [21]. In this investigation LME is therefore unlikely to occur. Other processes that may alter mechanical properties depend on the purity of the environment, i.e., the chemical activity of oxygen, nitrogen, carbon etc., in the liquid metal. In liquid sodium the oxygen potential is always very low, although, a sodium chromite

($NaCrO_2$) film may form on the metal surface in short times, this film is often spalls away during mechanical loading [22,23]. Sodium is unlikely to affect the strength and ductility of ferritic steel unless carbon movement occurs. The test duration of ~24 h is apparently insufficient to cause any significant decarburization [15]. Indirect effects could arise as a result of thermal ageing, but ageing at appreciably lower temperatures than the tempering temperature has little effect on tensile properties and hence unlikely to affect the fatigue behaviour [24].

The increase in LCF life in Na environment compared to air environment, can be evaluated with two contributions to overall fatigue life – i.e., crack initiation life and crack propagation life. Metallographic investigation of the fracture surface of the samples tested in Na environment indicates that oxidation effects are virtually absent (Fig. 7(a)). Specimens are absolutely free of oxides and any other corrosion products. On the other hand, fatigue tests conducted in air (Fig. 7(b)) shows substantial oxidation that may influence cyclic deformation in several ways. Oxidation of the slip steps can prevent slip reversal, segregation of nonmetallic elements at grain boundaries or slip bands may accelerate cracking and strengthening of the surface due to oxide film that can cause strain accumulation and enhanced cavitation all of which can alter near-surface deformation behaviour and thus alter mechanical properties such as fatigue crack initiation and propagation [25]. The analysis on the longitudinal cut specimens reveal that the sodium tested samples are free from secondary cracks in contrast to the air tested samples with a numerous secondary cracks (Fig. 8(a) and (b)). Similar results were reported by Ebi and McEvily [1]
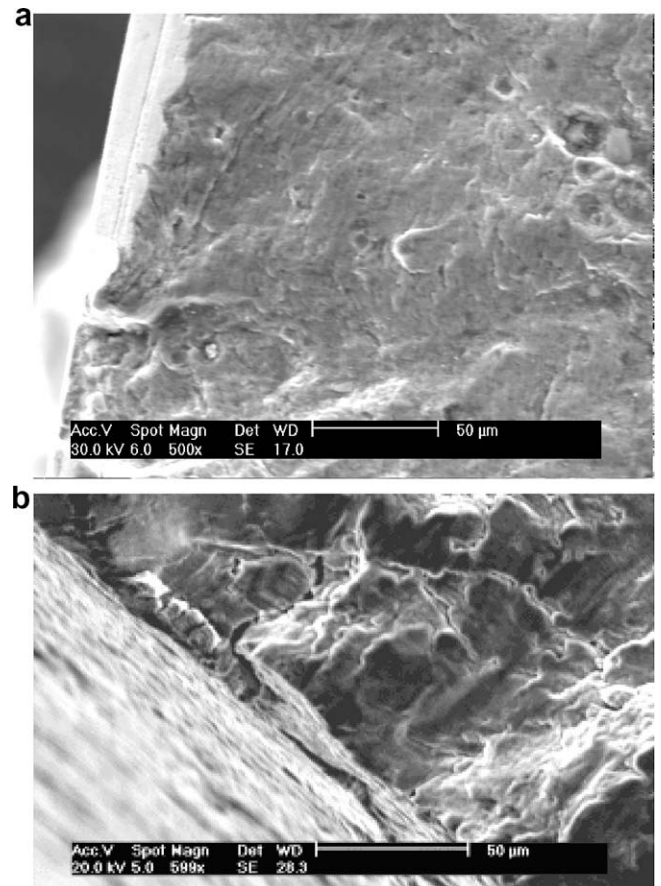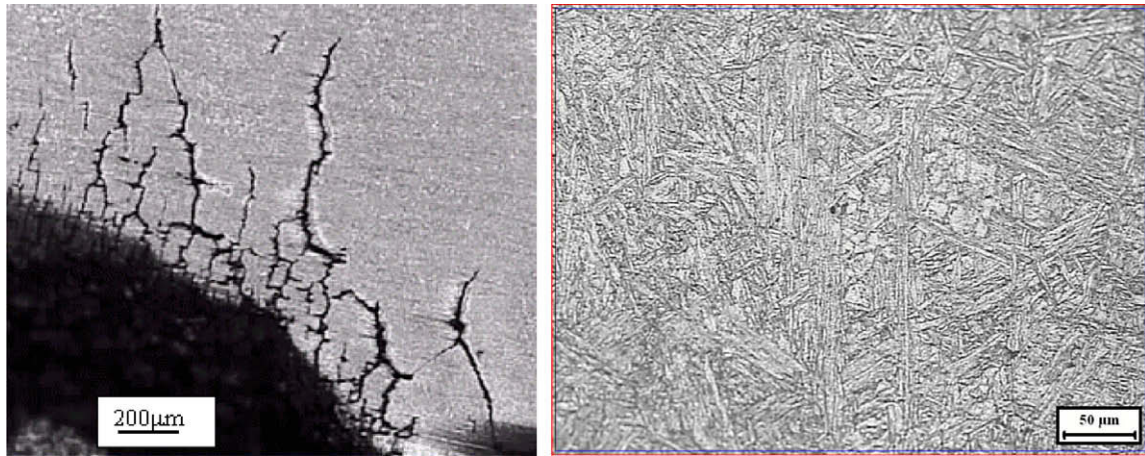

Fig. 7. (a) SEM picture depicting no oxidation of the fracture surface in sodium tested specimen (T = 823 K; strain amplitude ±0.4%; strain rate 3×10^{-3} s^{-1}) and (b) crack initiation assisted by oxidation under air environment (T = 873 K; strain amplitude ±0.6%; strain rate 3×10^{-3} s^{-1}).

Fig. 8. Cracking behaviour observed on the longitudinally cut specimen (a) large number of secondary cracks in air and (b) no secondary crack in sodium environment (T = 873 K; strain amplitude ±0.6%; strain rate 3×10^{-3} s^{-1}).

under vacuum environment. In Fig. 8(a), both along the specimen surface and at the crack tips, an internal layer, enriched in oxygen especially at lath and grain boundaries is observed. Once the oxide layer is cracked, a fresh metallic surface is directly in contact with the environment and oxidation starts by the penetration of oxygen along the fast diffusion paths (microstructural boundaries) [26]. These cracks of the oxide layer induce stress concentration at their tip in the material. Therefore it is easier for these cracks to propagate inside the material from these high-stress regions, than for a new crack to initiate in the bulk of the material somewhere else. Several authors [1,5,10] have attributed the reduction of fatigue life under oxidizing atmosphere to the reduction in crack initiation life. The slope of the strain–life plots (Figs. 5 and 6) are steeper at air atmosphere is an indication that the proportion of life time spent in crack initiation is less [1].

It has been postulated [27] that the decrease in crack propagation rate is due to lack of oxidation of the newly created crack surfaces during the tensile part of the cycle and partial rewelding during the compression part of the cycle. Crack growth is due to the applied mechanical load and oxidation effects due to oxygen penetration at the crack tip [28]. Environment modifies both the stress intensity threshold and the propagation rate above this threshold [29]. There is a continuous contribution of oxidation to the growth per cycle and the rate of fatigue crack growth cannot

be a single valued function of the stress intensity factor but must to some extent also depend on the time of exposure [29]. Based on the striation spacing measurement Sandhya et al. have demonstrated on 316L(N) SS [30] that the fatigue crack propagation rate was much lower in sodium environment. In case of high purity low oxygen sodium there will be virtually no contribution from the oxidation at the crack tip and hence the crack growth rate will be lower.

The lack of oxidation in sodium environment is responsible for delayed crack initiation and decreased crack propagation rate which in turn increases the fatigue endurance. Crack initiation being the dominant phase at lower strain amplitudes, the increase in life under Na environment is more pronounced at lower strain amplitudes (Figs. 5 and 6 and Table 2).

The data generated in this investigation is compared with RCC-MR [31] design curve for modified 9Cr–1Mo steel at 773 K in air environment (Fig. 9). The RCC-MR design curve values are multiplied by factors of two on strain and 20 on cycles and maximum thus obtained is taken as the best-fit curve. A statistical factor of ±25% was used in this calculation to derive the lower and upper bound curves. The data presented in this investigation lie above the upper bound curve of the air data. These results clearly indicate that design against LCF using data from in air tests is conservative.

4. Conclusions

LCF tests conducted in flowing sodium environment at 823 K and 873 K have shown that the evolution of cyclic stress response is similar to that of air environment. LCF lifetimes are significantly increased in sodium environment when compared to the data obtained in air environment. The lack of oxidation in sodium environment is considered to be responsible for delayed crack initiation and a lower crack propagation rate with a consequent increase in fatigue life. Crack initiation being the dominant factor at lower strain ranges, this improvement in life is more pronounced at lower strain ranges. Comparison of data obtained in sodium environment with RCC-MR design code data shows that design curve based on air tests is too conservative.
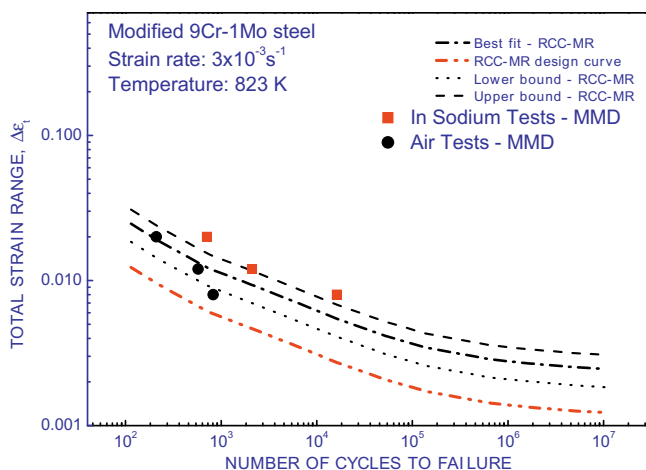
Fig. 9. Comparison of data with RCC-MR code predictions for modified 9Cr–1Mo steel.

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