PREFACE

The advanced power generating industries use high operating steam temperatures and pressures, to achieve higher efficiency. This led to development of alloys with superior properties at elevated temperatures. The two alloys under investigation are modified 9Cr-1Mo steel and Inconel 617 alloy. From the application point of view, the two alloys are used as piping and tubing materials in various components such as steam generator, super heater, re heater and heat exchanger etc. of power plants. Modified 9Cr-1Mo steel (grade 91) with strengthening elements such as Mo, V and Nb added to conventional 9Cr-1Mo steel possesses excellent mechanical properties, microstrutural stability at elevated temperatures and resistance to corrosion in comparison to low Cr-Mo steels, conventional 9Cr-1Mo and austenitic stainless steels. Addition of Cr improves the corrosion resistance and mechanical properties. Mo is a solid solution strengthener and is considered as a retardant for dislocation recovery/recrystallization. In this alloy, strength in normalized and tempered condition is derived from carbides like NbC, VC and $M_{23}C_6$ on sub-boundaries and from tempered martensitic laths with high dislocation densities. The other alloy under investigation is Inconel 617 alloy, which is a nickel based superalloy containing Cr, Co, Mo, Al and Ti as alloying elements. It is widely used in, high temperature applications because of its superior creep resistance and improved stability of microstructure for long exposures at elevated temperatures. A protective layer of Cr and Al oxides, forms on the surface of the alloy at elevated temperature, enhances its oxidation resistance. This tungsten free alloy is lighter and cost competitive as compared to other nickel based alloys with tungsten. It is primarily strengthened by, precipitation of fine homogeneously dispersed γ' phase in γ matrix and from precipitation of M_6C and $M_{23}C_6$ carbides, both at grain boundaries and in the grains.

Failure occurring under cyclic loading is termed as fatigue. The loading condition may change from symmetric to asymmetric. In low cycle fatigue, there is symmetrical loading (R=-1); therefore, plastic strain does not accumulate, whereas in asymmetrical loading hysteresis loops do not close, and plastic strain increases due to imposition of positive mean stress and this process is termed as ratcheting or cyclic creep. Due to progressive increase in tensile permanent deformation, cyclic life is drastically reduced, in respect of the low cycle fatigue. During service, components such as pipes and tubes in various industries and power plants undergo ratcheting due to fluctuation in internal pressure, temperature and seismic events; therefore, during the last three decades ratcheting fatigue has gained much importance. Various investigations have been conducted on different structural materials (e.g. different grades of steel, alloys of aluminium, copper, zirconium, magnesium, polymers etc.) in the area of ratcheting fatigue. However, the area of ratcheting under various parameters along with microstructural correlation is still unexplored, for the two alloys under study. The present investigation, aims to investigate the ratcheting fatigue behaviour of modified 9Cr-1Mo steel and Inconel 617 alloy, under various parameters (stress amplitude, mean stress, stress rate and temperature) along with deformation and fractography in detail. The thesis is divided into seven chapters.

Chapter 1- presents introduction of ratcheting fatigue and literature related to it, for various structural materials under different areas of experimentation, microstructural characterization and modelling. The alloys investigated in this study are modified 9Cr-1Mo steel and Inconel 617 alloy. There is brief introduction of both the alloys regarding design, development, physical metallurgy and applications. The need and importance of the present investigation is emphasized along with objectives of the study.

Chapter 2- presents details of materials and experiments carried out in the present investigation. Modified 9Cr-1Mo steel was obtained in normalized and tempered condition, in the form of 25 mm plate thickness whereas Inconel 617 alloy was procured as forged rod of 14 mm diameter and was solution annealed at 1175°C for 40 minutes and quenched in water. For finalizing the matrix for ratcheting fatigue experiments, tensile tests were conducted for both the alloys at a strain rate of 10^{-3} s^{-1} and at different temperatures. Ratcheting fatigue experiments were performed under asymmetrical loading, in stress controlled mode, for modified 9Cr-1Mo steel and Inconel 617 alloy under various parameters such as stress amplitude, mean stress, stress rate and temperature.

Fracture surfaces of the tensile and fatigue tested specimens were examined by scanning electron microscope. Deformation behaviour was analyzed using transmission electron microscope.

Chapter 3- deals with ratcheting fatigue behaviour of modified 9Cr-1Mo steel at room temperature. The effect of mean stress, stress amplitude and stress rate on ratcheting behaviour of modified 9Cr-1Mo steel was analyzed. It was observed that with increase in mean stress and stress amplitude, the accumulation of ratcheting strain increases whereas fatigue life decreases. Contrarily, with rise in stress rate, ratcheting strain decreases and there is enhancement in fatigue life, for the alloy investigated. On examining the fracture surface, a unique type of fracture was observed in case of modified 9Cr-1Mo steel with two distinct regions: the central region of the fracture surface showing dimples and the circumferential region depicting striations like features. Microstructural characterization on deformation under ratcheting showed that, in case of Modified 9Cr-1Mo steel the lath martensitic microstructure transformed into equiaxed subgrain structure (recovery).

Chapter 4- discusses the ratcheting fatigue behaviour of Inconel 617 alloy at room temperature. The effect of mean stress, stress amplitude and stress rate on ratcheting behaviour of Inconel 617 alloy was analyzed. Ratcheting strain increases with increase in mean stress and stress amplitude and fatigue life reduces whereas inverse behaviour was observed with rise in stress rate. Inconel 617 alloy exhibited typical fatigue fracture with striations. Microstructural characterization following deformation under ratcheting, exhibited slip bands at regular intervals.

Ratcheting fatigue behaviour of both the alloys at room temperature was compared with the help of normalized mean stress and stress amplitude at a constant stress rate of 150 MPa/s. It was observed the accumulation of ratcheting is very high for Inconel 617 alloy in comparison to modified 9Cr-1Mo steel. Inspite, of such high accumulation of plastic strain in Inconel 617 alloy, it still exhibited higher fatigue life. The reason for observed behaviour is higher value of uniform elongation and degree of work hardening in Inconel 617 alloy. Thus, uniform elongation can be considered as an important tensile parameter for selection of material for piping and tubing components, experiencing ratcheting fatigue.

Chapter 5- presents the ratcheting fatigue behaviour of modified 9Cr-1Mo steel and Inconel 617 alloy at their respective service temperature, under the influence of mean stress. The service temperature for modified 9Cr-1Mo steel is 600^{o} C whereas for Inconel 617 alloy it ranges between 750^{o}C- 850^{o}C; therefore it is taken as 800^{o} C, in the present investigation. With increase in mean stress the ratcheting strain increases, and there is reduction in fatigue life, for the two alloys under study at their respective service temperatures. Apart from this, the ratcheting behaviour of the two alloys at ambient temperature has been compared with their respective service temperatures. Hence, for the purpose of comparison the two parameters i.e. the stress amplitude and mean stress were normalized, with respect to tensile strength of the alloys, at that particular temperature, and the third parameter of stress rate was kept constant at 150 MPa/s. The comparison showed that for both the alloys, with increase in temperature, ratcheting strain decreases and there is improvement in the cyclic life of the alloy. The enhancement in fatigue life of the alloys at their respective service temperature may be attributed to modulus of elasticity and recovery processes. The fracture surfaces showed similar type of features as observed at room temperature for the two alloys under investigation. On examining the microstructures of the samples, deformed under ratcheting at elevated temperature, recovery along with recrystalliztion was observed for modified 9Cr-1Mo steel whereas there was formation of precipitates ($M_{23}C_6$) in Inconel 617 alloy.

The next part of the chapter deals with, ratcheting behaviour of the alloys at identical homologous temperature. The best way to compare the mechanical properties of different materials, at elevated temperatures is in terms of the ratio of the test temperature to the melting point, expressed in degrees Kelvin. This ratio is often referred to, as homologous temperature. This ratio in the present study, figures out to be 0.42 T_m. Ratcheting fatigue tests were conducted at 0.42 T_m with normalized mean stress, stress amplitude and at a constant stress rate of 150 MPa/s to study the behaviour of both the alloys. While comparing the behaviour of the two alloys it was observed that ratcheting strain accumulation was tremendously high for Inconel 617 alloy as compared to modified 9Cr-1Mo steel. The ratcheting curve shows steps at various intervals in case of Inconel 617 alloy whereas the curve is highly stable for modified 9Cr-1Mo steel. The abrupt increase in plastic strain accumulation, in Inconel 617 alloy may be attributed to unpinning effect of dislocations, pinned due to dynamic strain aging. Modified 9Cr-1Mo steel exhibits striations like features on smooth circumferential region and dimples in the central region of the fracture surface whereas Inconel 617 alloy depicts purely fatigue signature (striations). The deformation behaviour of modified 9Cr-1Mo steel at 0.42 T_m exhibits dislocation pile-ups, at boundaries along with precipitates which may provide barriers to the mobility of dislocations, on the other hand tangles of dislocations along with cross slipping was observed for Inconel 617 alloy.

Chapter 6- deals with the influence of ratcheting on tensile properties of modified 9Cr-1Mo steel and Inconel 617 alloy under the impact of mean stress, stress amplitude and stress rate at ambient temperature. The strength parameters increased while ductility decreased for ratcheted specimens as compared to the un-ratcheted specimen. With increase in mean stress and stress amplitude, the strength parameters increased while the ductility decreased for the two alloys under investigation. This can be very well explained with the cyclic hardening behaviour of the alloys under these two parameters.

Inversely, with increase in stress rate the strength parameters decreased while ductility increased and this behaviour is mainly attributed to the cyclic softening behaviour of the alloys. Inconel 617 alloy showed tremendous increase in yield strength of about 186% in comparison to modified 9Cr-1Mo steel. While examining the fracture surface, modified 9Cr-1Mo steel exhibited rosette fracture for un-ratcheted specimen, and for ratcheted specimen distinct regions have been observed, central region showing dimples and the circumferential region depicting striations like feature. Inconel 617 alloy showed purely ductile fracture with dimples on fracture surface, for ratcheted as well as un- ratcheted specimens. Microstructural characterization to observe the basic difference in deformation due to pre-ratcheting and to see the effect of the parameters was performed by transmission electron microscopy.

Chapter *7*- summarizes the major conclusions of the present investigation and scope for future work.