PREFACE

Modified 9Cr–1Mo steel, with minor additions of V and Nb in conventional 9Cr–1Mo steel, is widely used for steam generators of nuclear reactors. It is also a candidate material for future sodium cooled fast breeder reactors. It possesses higher thermal conductivity, lower thermal expansion coefficient, higher tensile and creep strength, better weldability, higher microstructural stability and resistance to stress corrosion cracking in water–steam systems in comparison to those of low Cr–Mo steel, conventional 9Cr–1Mo steel and austenitic stainless steels. During service, the components of steam generators are often subjected to repeated thermal stresses as a result of temperature gradients arising from heating and cooling during start–up and shutdown operations. These transient thermal stresses induce cyclic strains and cause low cycle fatigue (LCF) damage to material. LCF at elevated temperatures is known to be influenced by time dependent processes like dynamic strain ageing (DSA), creep, oxidation and microstructural degradation. DSA has been found deleterious to tensile ductility, LCF life and creep resistance of structural components.

In this thesis a detailed investigation is presented on DSA, deformation and fracture behavior of modified 9Cr–1Mo steel under tensile and cyclic loading, with an emphasis on the effects of DSA on dislocation substructure and cyclic stress response. The thesis comprises of two major parts (1) tensile bahvior including DSA, deformation and fracture (2) LCF behavior at room temperature (RT), 300 °C (DSA region) and 600 °C at strain rates of 10^{-2} s^{-1} & 10^{-3} s^{-1}.

The thesis is divided into eight chapters. Chapter 1 gives brief account of energy scenario in India and the importance of nuclear energy. It further gives a picture of the indigenous three stage nuclear energy programme and the important role of FBRs in this programme. The selection criteria of structural materials for different components of the

FBRs are also described. Development of modified 9Cr–1Mo steel, significance of the present study and the literature related tensile and fatigue deformation of modified 9Cr–1Mo steel is presented.

Chapter 2 provides the details of the material and the experimental methods used for the tensile test, low cycle fatigue test, internal friction test and study of deformation and fracture behavior.

DSA behavior of the modified 9Cr–1Mo steel under monotonic loading was studied at different strain rates from 10^{-3} to 10^{-5} s^{-1} between RT to 600 °C and presented in Chapter 3. Variation of tensile properties with temperature exhibited a plateau/peak in yield, tensile strength, minima in ductility, serrations in stress–strain curves and negative strain rate sensitivity from 250 to 400 °C. These characteristic features point to occurrence of DSA. There was also Snoek peak in internal friction. Activation energy for the process of DSA was found to be 58 kJ/mol, which match with activation energy for diffusion of nitrogen in iron. Thus, DSA in this steel may be attributed to pinning of dislocations by nitrogen atoms. Irrespective of test temperature from 200–450 °C, there was formation of dislocation cell structure. There was high density of dislocations in the regime of DSA and features like dislocation debris, kinks and bowing of dislocations and decrease in dislocation cell size were observed.

In general, ductile metallic materials exhibit typical cup and cone fracture with central fibrous zone and shear lip zone. However, the modified 9Cr–1Mo steel exhibited rosette fracture, comprising of fibrous zone along with radial cracking resulting from longitudinal splitting parallel to the loading axis. Chapter 4 deals with detailed investigation on the process of rosette fracture in the modified 9Cr–1Mo steel. The rosette was influenced by the test temperature (< 100 °C), microstructure (in particular with number density, size and distribution of carbide precipitates along the prior austenite grain boundaries/lath boundaries), and plastic constraint around the crack. No effect of material texture was observed on rosette fracture occurring in this steel. The process of rosette fracture was found to be associated essentially with void nucleation at carbide particles lying on the prior austenite grain boundaries as well as on lath boundaries, rapid growth and linkage of voids preferentially along the grain boundaries oriented parallel to stress axis.

Chapter 5 describes the LCF behavior of modified 9Cr–1Mo steel at RT. This steel exhibited initial mild hardening followed by continuous softening till failure. On the other hand, at the lower strain amplitudes (≤ ±0.31%) there was stabilized stress response up to the initial 30 cycles, followed by mild hardening and subsequent continuous softening. The rate of softening increased with increase in strain amplitude. The cyclic stress response was found to decrease with decrease in strain rate. A linear dependence of LCF life was observed with both plastic strain amplitude as well as strain energy. Fatigue life, however, was found to reduce with decrease in strain rate, in particular at low strain amplitudes. Analysis of cyclic stress–strain hysteresis loops exhibited Masing behavior at higher strain amplitudes (≥ ±0.375%) and non–Masing behavior at lower strain amplitudes (< ±0.375%) at both the strain rates. Friction stress was found to decrease with number of cycles for all the strain amplitudes studied whereas the back stress remained nearly constant at higher strain amplitudes and increased at the lowest strain amplitude. Deformation study revealed transformation of tempered lath martensitic structure into equiaxed cell structure at low strain amplitude and to elongated cell structure at high strain amplitudes. Thus, the observed cyclic softening was mainly due to decrease in friction stress, formation of low energy dislocation cell structure and annihilation of dislocations. The initial hardening in the cyclic stress response may be attributed to work hardening. It has been established that the transition from non–Masing to Masing behavior is mainly due to change in the microstructure/dislocations configuration. Thus, the transition of non–Masing to Masing behavior may be associated with change in dislocation structure from equiaxed cell to elongated cell.

Chapter 6 deals with LCF behaviour of modified 9Cr–1Mo steel at 300 °C. Cyclic stress response at 300 °C was found to be similar to that at RT, exhibiting initial mild hardening and subsequent cyclic softening till failure. However, an inverse effect of strain rate was observed on CSR, fatigue life and plastic strain amplitude. There was increase in cyclic stress response, decrease in fatigue life and reduction in plastic strain amplitude with decrease in strain rate. These features reflected occurrence of DSA in the modified 9Cr–1Mo steel under cyclic strain loading at this temperature. Influence of DSA was observed also on the resulting dislocation substructure and fracture behavior. Bowing of dislocations and dislocation cell structure was observed at all the strain amplitudes. Interstriation spacing in stage II fatigue crack was found to increase with increase in strain amplitude and was higher at lower strain rate. It is important to mention that there was transformation of equiaxed cell structure occurring at low strain amplitude of ±0.25%, to elongated cell structure at the higher strain amplitude of ±0.375% and also there was transition of Masing behavior to non–Masing behaviour with increase/decrease of strain amplitude; similar to that at RT.

LCF behaviour at 600 °C is presented in chapter 7. Cyclic softening at all the strain amplitudes and strain rates was observed to be similar to that at RT and 300 °C, however, with higher rate of softening. Unlike the behavior at 300 °C the cyclic stress response was reduced with decrease in strain rate and there was lower fatigue life at the strain rate of 10^{-3} s^{-1}. It is essential and important to mention that there was oxidation of the steel at 600 °C at all the strain amplitudes and strain rates. The severity of oxidation was increased with decrease in strain rate. Thus, decrease in fatigue life at low strain rate was associated with oxidation effect. Thus, cyclic softening in this steel at 600 °C could be associated with different factors like cell formation, coarsening of carbides, dynamic recovery/recrystallization, annihilation of array of dislocations and grain rotation.

Chapter 8 summarizes all the important findings and presents and presents the scope of further investigation.