Chapter 2

Materials and Methods

2.1 Introduction

This chapter gives the chemical composition of the alloys selected for the present investigation along with the details of heat treatment, mechanical testing and microstructural characterization. Specimen preparation, geometry of specimens used for tensile and fatigue testing and specific equipments used for testing are also discussed. Metallographic procedures for optical microscopy, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are explained in detail.

2.2 Materials

In the present, study two different alloys i.e. modified 9Cr-1Mo steel and Inconel 617 alloy were investigated. Modified 9Cr-1Mo steel was received from IGCAR, Kalpakkam, Tamil Nadu, in the form of 25 mm thick plates. The alloy was initially in the form of an ingot of 600 mm diameter and was forged into a slab of cross section 95 mm x 350 mm, in the temperature range from 1100 to 800^{o}C. The slab was successively subjected to hot rolling in the temperature range from 1050 to 800^{o}C to produce a plate of cross section 25 mm x 400 mm. The hot rolled plate was subjected to further heat treatment. Normalizing was carried out by heating at 1060^{o}C for 1 h, followed by cooling to room temperature in air and subsequent tempering at 780^{o}C for 1 h and cooling to room temperature in air. Table 2.1 gives chemical composition of the present steel.

Table 2.1 Chemical composition of the modified 9Cr-1Mo steel (weight %)

Fe	Cr	Mo	Mn	Ni	Si	V	C	Nb	N	P	Al	S
Bal	9.27	0.95	0.41	0.33	0.38	0.21	0.1	0.074	0.044	0.018	0.013	0.002

Inconel 617 alloy was procured from M/s. Bharat Aerospace Metals Mumbai, in the form of 14 mm diameter rod in hot forged condition. Hot forging was carried out in the temperature range of 900-1100°C. These rods were subjected to solution treatment at 1175°C for 40 minutes and quenched in water. The chemical composition of the alloy is shown in Table 2.2

Table 2.2 Chemical composition of the Inconel 617 alloy (weight %)

Ni	Cr	Co	Mo	Al	Ti	Fe	Si	C	B	Mn	Cu	S
Bal	20.61	11.11	8.92	0.81	0.38	0.77	0.26	0.066	0.002	0.019	0.023	<0.003

2.3 Mechanical Testing

2.3.1 Tensile Testing

Cylindrical tensile specimens were machined of the two alloys, with gauge length and diameter of 15.5 mm and 4.5 mm, respectively. A schematic geometry of the tensile specimen used in the investigation is shown in Fig. 2.1. Gauge section of the tensile specimen was mechanically polished to remove machining marks and scratches. Tensile tests were carried out using screw driven universal testing machine of 100 kN load capacity (Instron Model No. 5982), at room temperature, at a strain rate of 10^{-3} s^{-1}. A room temperature extensometer (Model: Instron 2630-102) with gauge length of 10 mm was used for measuring extension.

High temperature tests were conducted using the same machine with the help of a single zone resistance heating furnace. Specimens were kept for 15 minutes at the desired temperature before starting the test. Tensile tests were also performed on the fatigue specimens, pre-ratcheted for 200 cycles at room temperature as well as high temperatures at a strain rate of $10^{-3}s^{-1}$.

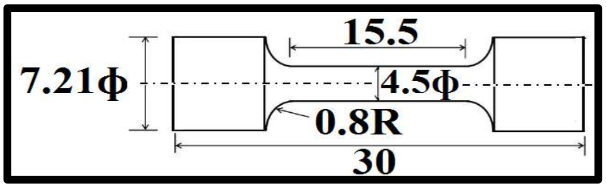

Fig. 2.1 Geometry of the cylindrical tensile specimen.

2.3.2 Ratcheting Fatigue Testing

Cylindrical fatigue test specimens were prepared for studying the ratcheting fatigue behaviour, with gauge section of 5.5 mm diameter, 15 mm length and threaded ends of M x 12 x 35 mm length (on either side), and radii of curvature of 25 mm. Fig. 2.2 depicts the geometry of specimens used for ratcheting fatigue testing. Gauge sections of the test specimens were polished with emery papers of 1/0 to 4/0 grades and finally with alumina powder.

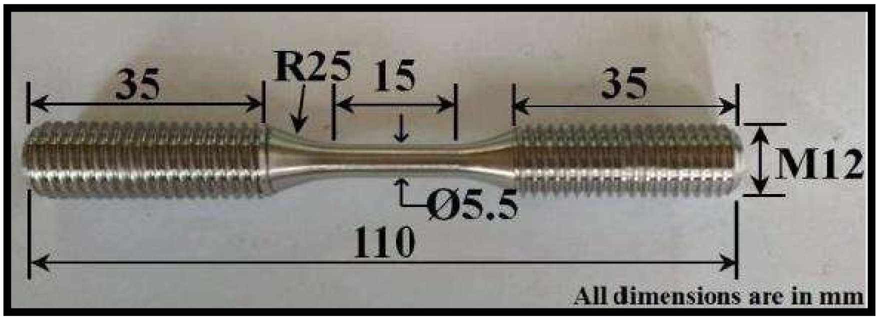

Fig. 2.2 Schematic of cylindrical fatigue specimen.

Ratcheting fatigue tests for the alloys were performed under asymmetrical stress controlled mode with various test parameters. The parameters used are defined as follows:

- **SYMMETRICAL LOADING:** The stress levels (maximum and minimum) are equal in tension and compression during cycling (R=-1).

$$\boldsymbol{Stress\ ratio\ (R)} = \frac{\sigma_{min}}{\sigma_{max}} \tag{2.1}$$

- **ASYMMETRICAL LOADING:** The stress levels (maximum and minimum) are unequal in tension and compression during cycling ($R \neq -1$).

- **STRESS AMPLITUDE:** One half of the algebraic difference between the maximum and minimum stress.

$$\boldsymbol{Stress\ amplitude\ (\sigma_a)} = \frac{(\sigma_{max}-\sigma_{min})}{2} \tag{2.2}$$

- **MEAN STRESS:** Arithmetic mean of maximum and minimum stress.

$$\boldsymbol{Mean\ Stress\ (\sigma_m)} = \frac{(\sigma_{max}+\sigma_{min})}{2} \tag{2.3}$$

- **STRESS RATE:** The rate at which stress is applied.

$$\boldsymbol{Stress\ rate\ (\dot{\sigma}) = 4 \times frequency \times \sigma_a} \tag{2.4}$$

- **NORMALIZED MEAN STRESS:** For comparison purpose, the mean stress used for one alloy is normalized with respect to ultimate tensile strength (σ_{UTS}) so that the ratio becomes constant for both the alloys.

$$\boldsymbol{Normalised\ mean\ stress\ \ \sigma_{nm}} = \frac{\sigma_m}{\sigma_{UTS}} \tag{2.5}$$

- **NORMALIZED STRESS AMPLITUDE:** For comparison purpose, the stress amplitude used for one alloy is normalized with respect to ultimate tensile strength (σ_{UTS}) so that the ratio becomes constant for both the alloys.

$$\boldsymbol{Normalised\ stress\ amplitude\ \ \sigma_{na}} = \frac{\sigma_a}{\sigma_{UTS}} \tag{2.6}$$

- **SERVICE TEMPERATURE:** The temperature at which the alloys are used in service. For modified 9Cr-1Mo, the service temperature is 600^{o} C whereas for Inconel 617 alloy, it is 800^{o} C.
- **HOMOLOGUS TEMPERATURE:** The ratio of the test temperature to the melting point, expressed in degrees Kelvin, is known as the homologous temperature.

Uniaxial ratcheting fatigue tests were conducted for both the alloys, with triangular waveform as depicted in Fig. 2.3 under different combinations of mean stress, stress amplitude and stress rate at room and elevated temperatures, with one parameter as variable and keeping the other two parameters constant.

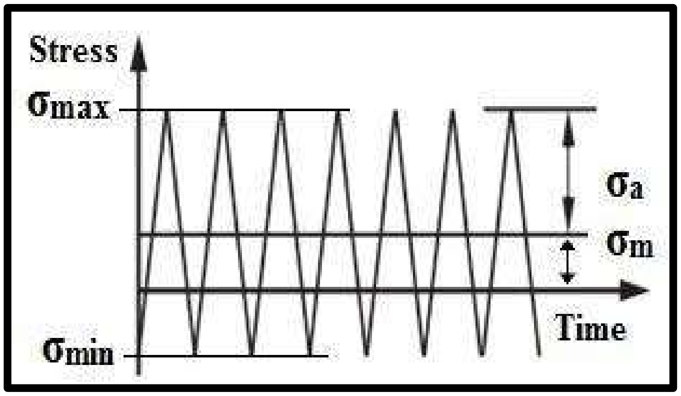

Fig. 2.3 Schematic of asymmetric stress controlled cyclic loading, where stress ratio ($R \neq -1$), mean stress ($\sigma_m \neq 0$) and stress amplitude ($\sigma_a \neq 0$).

While comparing the ratcheting behaviour of the respective alloy at ambient and service temperature and of the two alloys at homologous temperature, the mean stress and stress amplitude were normalized with respect to their ultimate tensile strengths at that particular temperature, at a constant rate of stressing. These experiments were conducted using a 50 kN servo hydraulic MTS (Model 810) equipped with fully automatic flex test 40 controller as shown in Fig. 2.4.

The plastic strain accumulation and displacements with applied peak and valley stresses both in tensile and compressive part of each cycle were stored and displayed using the controller software. A high temperature extensometer (Model: MTS- 632.53E) of 12 mm gauge length was mounted over the gauge section of the specimen, at all the test temperatures studied.

Fig. 2.4 Servo-hydraulic Material Testing System (MTS).

In case of high temperature testing, a three zone resistance heating furnace was used for heating the samples. The experimental set-up for high temperature fatigue testing along with furnace is shown in Fig. 2.5(a). Uniform heating of the specimens was accomplished with the help of three separately attached thermocouples (one at gauge section and two at both ends) and by carefully monitoring and controlling the temperature. The high temperature extensometer along with the thermocouples was mounted on the sample as shown in Fig. 2.5(b). The temperature gradient along the gauge section was controlled within ±2^{o}C of the required temperature. The specimens were soaked at the test temperatures for 15 minutes for homogeneity of temperature over the gauge section before applying the load.

Fig. 2.5 (a) Machine set up for high temperature ratcheting fatigue experiments and (b) High temperature extensometer and thermocouples mounted on the sample.

2.4 Microstructural Characterization

2.4.1 Optical Microscopy

In case of the modified 9Cr-1Mo steel, for initial characterization of the material, a cubic piece of 10 mm x 10 mm x 10 mm size was prepared from the normalized and tempered plate whereas for the Inconel 617 alloy, a cylindrical sample of 12 mm diameter and 10 mm length was sectioned from the solution treated rod for optical microscopy. The specimens were ground on both sides to make the sample flat, using belt emery grinder. The samples were then mechanically polished employing different grades of emery paper from 1/0 to 4/0 and finally polished on sylvet cloth using slurry of alumina powder and water on disc polisher. The polished surface was etched with Villella's reagent (1 gm picric acid, 5ml concentrated HCl and 100ml ethyl alcohol) for modified 9Cr-1Mo steel. The etchant used for Inconel 617 was Aquaregia (75 ml HCl and 25 ml HNO_3).

Microstructure examination of the as-received and heat-treated samples was carried out using optical microscope (Model: Image Analyzer) at different magnifications.

2.4.2 Scanning Electron Microscopy

The initial characterization of both the alloys in heat-treated conditions was also carried out through field emission scanning electron microscope (Model: FEI Quanta 200 F) operated at 30 kV (Fig. 2.6), using the sample prepared for optical microscopy.

Fracture ends of the tested specimens were transversely sectioned using slow speed circular saw for fractography. The fracture surfaces were ultrasonically cleaned with acetone and examined under scanning electron microscope. The fracture behaviour of tensile and fatigue tested specimens at room and high temperatures were analyzed, using scanning electron microscopy (SEM).

Fig. 2.6 Field emission scanning electron microscope (Model: FEI Quanta 200 F).

2.4.3 Transmission Electron Microscopy

Initial microstructural characterization of the heat treated alloys and detailed deformation analysis of the failed specimens of the two alloys, tested at ambient as well as elevated temperatures, was performed with the aid of transmission electron microscope (Model FEI Technai 20 G^2) operating at 200 kV (Fig. 2.7). Thin slices of 0.2 mm thickness were transversely sectioned from initial heat treated material and from gauge section of the fractured specimens with a slow speed diamond cutter to prepare TEM foils. These slices were mechanically thinned down to 50 microns and discs of 3 mm diameter were punched out of the thinned slices. Using twin jet electro polisher (Model: Struers Tenupol 5), the discs were electropolished in a suitable electrolyte to prepare TEM foils. The electrolyte taken for electropolishing in case of modified 9Cr-1Mo steel was 6% perchloric acid and 34% n-butanol in methanol, at 20V and -40°C whereas for the Inconel 617 alloy, 5% perchloric acid and 95% methanol and at 18V and -40°C was used for preparing TEM foils.

Fig. 2.7 Transmission electron microscope (Model FEI Technai 20 G^2).

2.5 Summary

This chapter briefly describes the alloys studied in the present investigation, specimen geometry, machines used for tensile and ratcheting fatigue testing. The metallographic procedures employed to obtain specimens for microstructural characterization are also presented.