Chapter 2
Material and Methods

2.1 Introduction

This chapter describes details of the processing and heat treatment of the material, modified 9Cr–1Mo steel, used in the present investigation. Test details like specimen preparation and geometry of tensile and low cycle fatigue (LCF) specimens as well as the testing parameters; temperature and strain rate for conducting tensile and LCF tests are included. Also the equipments used for testing and characterization in this investigation are mentioned.

2.2 Material

The material, modified 9Cr–1Mo steel, used in the present investigation was supplied by IGCAR, Kalpakkam, Tamilnadu, under the project funded by the Board of Research in Nuclear Sciences (BRNS), Department of Atomic Energy of India. The chemical composition of the modified 9Cr–1Mo steel is recorded in Table 2.1.

Table 2.1 Chemical composition of the modified 9Cr–1Mo steel in wt%.

Elements	**C**	**Si**	**Mn**	**P**	**S**	**Cr**	**Mo**	**Ni**
Wt. %	0.1	0.26	0.41	0.018	0.002	**9.27**	**0.95**	0.33

Al	**Nb**	**N**	**V**	**Fe**
0.013	**0.074**	0.044	**0.21**	Balance

The processing and heat treatments given to the material are shown in the flow chart in Fig. 2.1. The material in the form of ingot of 600 mm diameter was forged to a slab of 95 mm × 350 mm cross section in the temperature range from 1100 to 800 °C and the slab was successively subjected to hot rolling in the temperature range from

1050 to 800 °C to produce a plate of 25 mm × 400 mm cross section. The hot rolled plate was heat treated. Normalizing was carried out at 1060 °C for 1 h, followed by cooling in air to room temperature (RT) and subsequent tempering at 780 °C for 1 h and cooling in air to RT.

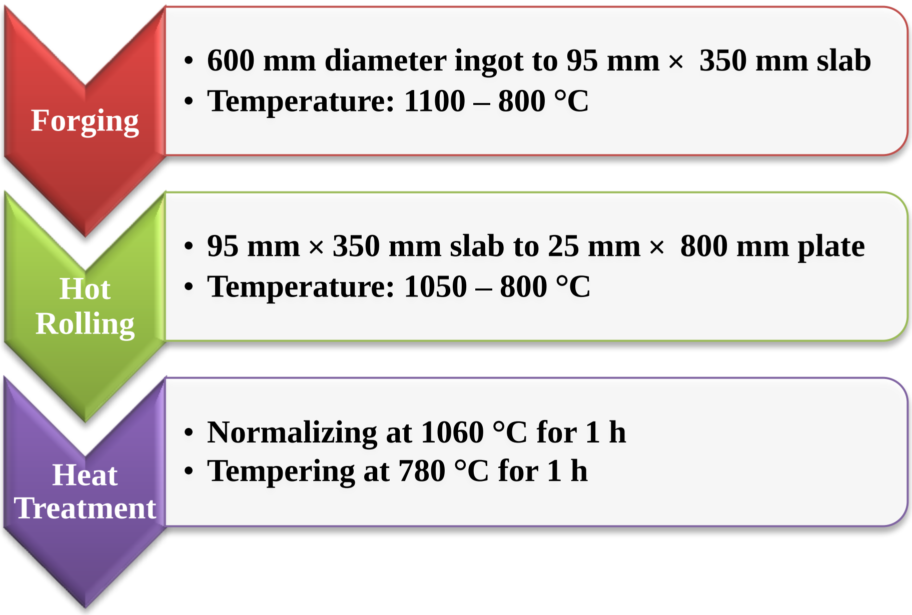

Fig. 2.1 Flow chart of the processing and heat treatment.

2.3 Microstructural Characterization

A cuboidal piece of 10 mm × 10 mm × 10 mm size was prepared from the normalized and tempered plate of the modified 9Cr–1Mo steel for optical and scanning electron microscopy. The specimen was grinded to make the opposite side surfaces flat and subsequently was mechanically polished on different grades of emery paper from 1/0 to 4/0 and finally on sylvet cloth using slurry of alumina powder and water. The polished surface was etched with Villella's reagent (1 gm picric acid, 5 ml conc. HCl & 100 ml ethyl alcohol) to reveal the microstructure. Microstructural examination was carried out using optical microscope (Model: Image Analyzer) and scanning electron microscope (Model: FEI Quanta 200F).

Detailed microstructural characterization was carried out using transmission electron microscope (Model: FEI Tecnai 20 G^2) operated at 200 kV. Thin slices of ≈ 0.5 mm thickness were sectioned from the normalized and tempered cuboidal piece using slow speed abrasive cutter, to prepare TEM foils. The thickness of the thin section was reduced to ≈ 50 μm mechanically and discs of 3 mm diameter were punched out. The thin discs of 3 mm diameter were electropolished in an electrolyte containing 6% perchloric acid and 34% n–butanol in methanol at 20V, cooled to -40 °C, using an twin jet electropolisher (Model: Struers Tenupol 5) to prepare TEM foil.

2.4 Mechanical Testing

2.4.1 Tensile Tests

Blanks of 40 mm × 15 mm × 10 mm size were sectioned from the as received (normalized and tempered) plate and cylindrical tensile specimens were machined with gage length and diameter of 15 mm and 4.5 mm respectively. Schematic of tensile test specimen is shown in Fig. 2.2.

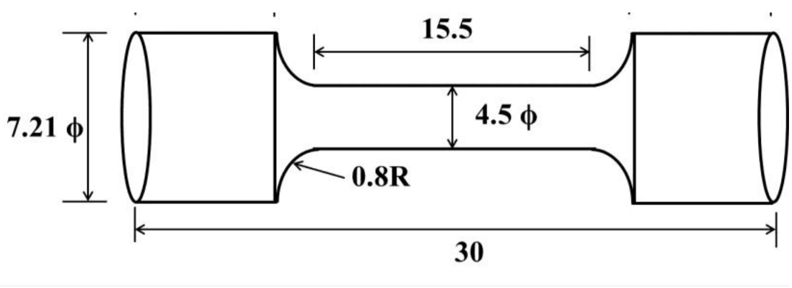

Fig. 2.2 Geometry of the cylindrical tensile specimen with dimensions in mm.

Gage section of the tensile specimens was mechanically polished to remove machining marks, if any. Tensile tests were performed on a 100 kN screw–driven Instron™ Universal Testing Machine (Model: 4206) over a wide range of temperature from RT to 600 °C, at nominal strain rates of 10^{-5}, 10^{-4} and 10^{-3} s^{-1}.

The test matrix is given in Table 2.2. In situ specimen heating to desired test temperature was achieved using a split electric resistance heating furnace. Before starting the tensile tests, specimens were soaked at test temperatures for 15 minutes to stabilize and homogenize the temperature of the specimen. Load elongation data were collected from the software.

Table 2.2 Test matrix for tensile tests to establish dynamic strain ageing behavior.

Strain Rate (s^{-1})	**Temperature (°C)**
10^{-3}	RT,200,250,300,350,400,450 & 600
10^{-4}	RT,200,250,300,350,400,450 & 600
10^{-5}	RT,200,250,300,350,400,450 & 600

A different set of tensile tests were performed to study rosette fracture. In general ductile materials exhibit characteristic cup and cone fracture and brittle materials display cleavage fracture under uniaxial tensile loading, however, the modified 9Cr–1Mo steel exhibited a typical rosette fracture in normalized & tempered condition under tensile loading at RT. In order to understand the process of rosette fracture, additional tensile tests were conducted in temperature range from -70 °C to

100 °C at a strain rate of 10^{-4} s^{-1} for the specimens, prepared from blanks oriented at 45 ° and 90 ° to the rolling direction. The schematic of specimen orientations is shown in Fig. 2.3. The effect of microstructure and also the strain rate was studied on the occurrence of rosette fracture in this steel.

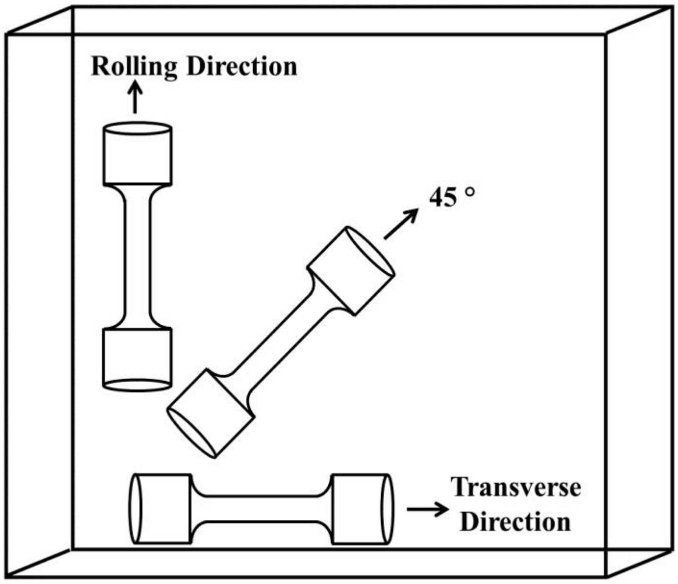

Fig. 2.3 Specimens fabricated from different orientations of blanks from the plate.

Different heat treatments were given to the as received (normalized and tempered) steel to bring out the effect of microstructure on rosette fracture and details of these tests are given in Table 2.3. The austenisation temperature was raised above that usually used, to dissolve the MX type carbide–nitride where M stands for metallic elements, and X for C and/or N, respectively. Macro–texture measurement was carried out for the different specimens using a Panalytical High Resolution X–Ray Goniometer (Model PW 3040/60).

Table 2.3 Details of different heat treatments given to the as supplied (normalized & tempered) modified 9Cr–1Mo steel.

Heat treatment	Designation
As supplied material (Normalized at 1060 °C for 1 h and tempered at 780 °C for 1h).	N_1+T
Annealing at 1100 °C for 2 h	A
Soaking at 1100 °C for 2 h, cooling to 1060 °C – soaking for 1 h and cooling in air.	N_2
N_2 specimen tempered at 780 °C for 1 h	N_2+T
Soaking at 1100 °C for 2h, cooling to 1060 °c – soaking for 1 h and quenching in water	S_2+WQ
S_2+WQ specimen tempered at 780 °C for 1 h	S_2+WQ+T

2.4.2 Low Cycle Fatigue Test

Cylindrical LCF specimens with gage section of 5.5 mm diameter, 15 mm length and threaded ends of 12 mm diameter & 35 mm length (on either side), and radii of curvature of 25 mm were machined with length in rolling direction of the plate (Fig.2.4).

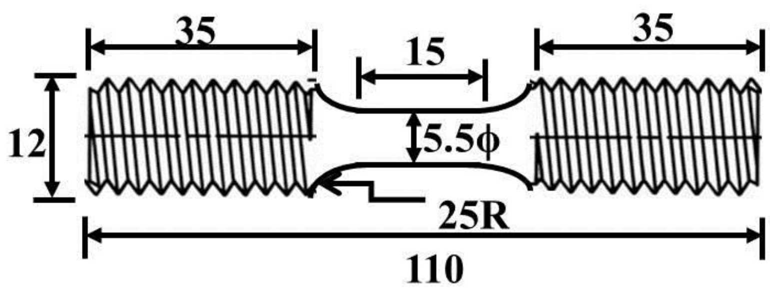

Fig. 2.4 Schematic of the cylindrical LCF specimen with dimensions in mm.

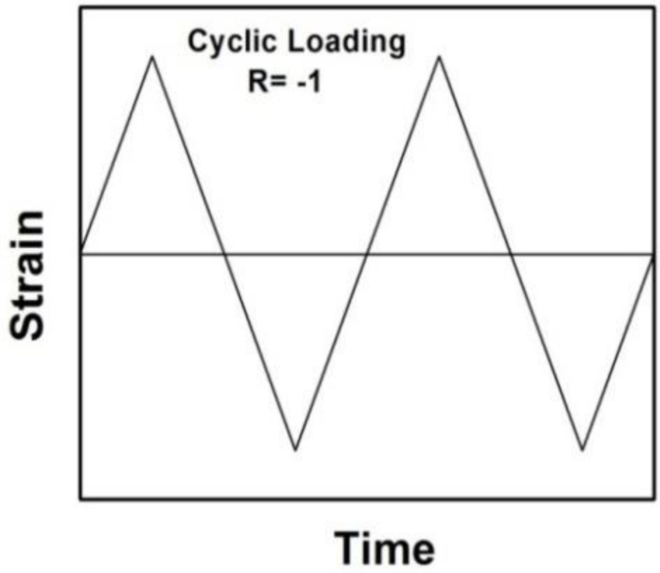

Fig. 2.5 Schematic of the reversed triangular waveform.

Strain controlled LCF tests were conducted under fully reversed triangular waveform (Fig. 2.5) in total–strain control mode using servo–hydraulic testing system (MTS model 810, with flex test 40 controller). LCF tests were performed at three different temperatures; RT, 300 °C and 600 °C in the regime of low strain amplitude at different strain rates of 10^{-2} s^{-1} and 10^{-3} s^{-1}. Test matrix for LCF is given in Table 2.4.

Table 2.4. Text matrix of LCF tests.

Test Temperature	Strain Amplitude	Strain Rate
RT	±0.25%, ±0.312%, ±0.375%, ±0.42%, ±0.50%	10^{-2} s^{-1} & 10^{-3} s^{-1}
300 °C	±0.25%, ±0.312%, ±0.375%, ±0.42%, ±0.50%	10^{-2} s^{-1} & 10^{-3} s^{-1}
600 °C	±0.25%, ±0.312%, ±0.375%, ±0.42%, ±0.50%	10^{-2} s^{-1} & 10^{-3} s^{-1}

2.4.3 Internal Friction Test

Internal friction was determined using inverted torsion pendulum shown in Fig. 2.6. The measurements were made over the temperature range from RT to 450 °C for the normalized and tempered and for 5% & 10% cold worked conditions. It was calculated by measuring the amplitude of oscillation with frequency. Two well defined peaks were observed in the plot of amplitude of oscillation vs frequency, from RT to 450 °C. It was calculated using the relationship

$$Q^{-1} = \frac{\Delta\omega}{\sqrt{3}\omega_0}$$

Where $\Delta\omega$ is full width at half maxima and ω_0 is angular frequency at maximum amplitude.

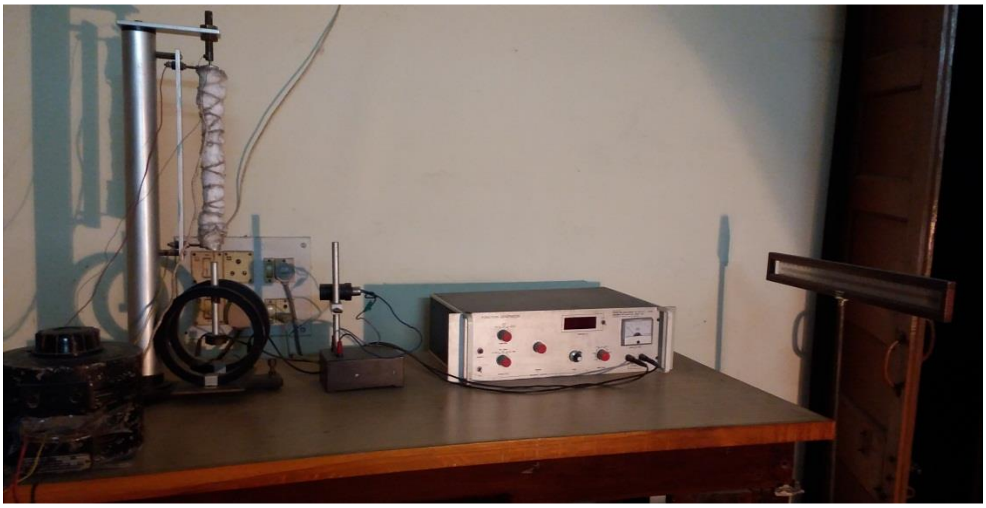

Fig. 2.6 Inverted torsional pendulum for internal friction measurement.

2.5 Microstructural Characterization of Tensile and LCF Tested Specimens

Fracture surfaces of the tensile and LCF tested specimens were transversely sectioned close to the fracture ends, using slow speed circular saw. Fracture surfaces were cleaned in ultrasonic cleaner with acetone. All the fractured surfaces were examined using field emission scanning electron microscope (FEI Quanta 200 F) to bring out the effect of temperature and strain rate.

The deformation behavior of tensile and fatigue tested specimen was characterized by TEM. The specimens were prepared as per the method mentioned in the section 2.3.