CHAPTER -2

Material and Methods

2.1 Introduction

The present chapter gives details of the material selected for the present study and heat treatment given along with the details of characterization and testing of the samples. Initial microstructural characterization, specimen geometry of the tensile and fatigue samples, equipment used for tensile and low cycle fatigue testing used for the present investigation are described in detail. The procedure for salt spray coating and thermal barrier coating is also mentioned.

2.2 Material

The material, Inconel 617 alloy used in the present study was supplied by M/s. Bharat Aerospace Metals, Mumbai in the form of 14 mm diameter rod in hot forged condition. It was given hot forging in the temperature range of 900 - 1100^{o}C. The chemical composition of the alloy is recorded in Table 2.1. Samples taken from the as received rods were solution annealed at 1175^{O}C for 40 minutes and quenched in water.

Table 2.1: 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 Initial Characterization

A cylindrical sample of 12 mm diameter and 10 mm length was sectioned from the solution annealed material for optical and scanning electron microscopy. The specimen was ground on both sides to make the sample flat at both ends using belt emery grinder. The samples were then mechanically polished using 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 Aquaregia

(75ml HCl and 25ml HNO_3) to reveal the microstructure. Microstructural characterisation was carried out using optical microscope (Model: Image Analyzer) and scanning electron microscope (Model: FEI Quanta 200F).

Detailed microstructural characterization of the initial material was carried out using transmission electron microscope (Model: FEI Tecnai 20 G^2) operating at 200 kV. Thin transverse slices of 0.2 mm thickness were sectioned from solution annealed samples with slow speed diamond cutter to prepare TEM foils. The thickness of the slice was reduced to thickness of 50 μm and discs of 3 mm diameter were punched out from the thinned slices. These were electro polished at 18V and -40°C, using a twin jet electro polisher (Model: Struers Tenupol 5). An electrolyte containing 5% perchloric acid and 95% methanol was used for the purpose.

2.4 Tensile Behaviour

2.4.1 Tensile Testing

To study the effect of temperature on tensile properties and establish DSA regime for the Inconel 617 alloy, cylindrical tensile specimens were machined with gage length and diameter of 15.5 mm and 4.5 mm respectively from the annealed material. Schematic of the tensile specimen used in the investigation is shown in Fig. 2.1. Gage section of the tensile specimens was mechanically polished up to 1000-grit surface finish using SiC paper to remove machining marks and scratches, if any. Tensile tests were performed using 100 kN screw–driven Instron™ Universal Testing Machine (Model: 4206) over a wide range of temperature from RT to 900°C, at strain rates of $5x10^{-4}$ s^{-1}, $5x10^{-3}$ s^{-1} and $1x10^{-2}$ s^{-1}. The test matrix for tensile tests to determine the DSA regime is given in Table 2.2. In situ specimen heating to the desired test temperature was achieved using a split electric resistance heating furnace. Before starting the tensile testing, specimens were soaked at test temperatures for 15 minutes to stabilize and homogenize the temperature of the specimen. Temperature was maintained within ± 5°C of the set point using a PID temperature controller.

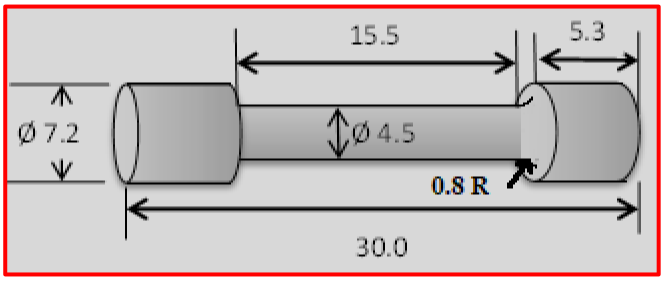

Fig. 2.1: Geometry of the cylindrical tensile specimen (Dimensions are in mm).

Table 2.2: Test Matrix for Tensile Testing.

Strain Rate (s^{-1})	**Temperature (°C)**
5×10^{-4}	RT, 200, 300, 400, 500, 600, 700, 800 & 900
5×10^{-3}	RT, 200, 300, 400, 500, 600, 700, 800 & 900
1×10^{-2}	RT, 200, 300, 400, 500, 600, 700, 800 & 900

2.4.2 Work Hardening Behaviour

To study effect of microstructure on work hardening behaviour of Inconel 617 alloy, a different test matrix was designed for carrying out tensile testing. Five different conditions of the alloy were prepared namely, the as received (AR), solution annealed and quenched (SQ), aged for 100h (SQ-AG1), aged for 500h (SQ-AG2) and cold worked (CW) conditions. The solution treated alloy was further aged at 850°C for 100h (SQ-AG1) and 500h (SQ-AG2) and cooled in the air. The solution treated rod of 14mm was cold rolled in four passes to reduce the cross section area by 40% to investigate the effect of cold working (SQ-CW). Various conditions of the alloy along with their designations are listed in Table 2.3.

Table 2.3: Different Conditions and Designations of the Inconel 617 Alloy.

S. No.	Condition	Designation
1	As received- hot forged in the temperature range 900-1100^{o}C	AR
2	Solution treated at 1175^{o}C for 40 minutes and water quenched	SQ
3	Solution treated at 1175^{o}C for 40 minutes, water quenched and aged at 850^{o}C for 100h	SQ-AG1
4	Solution treated at 1175^{o}C for 40 minutes, water quenched and aged at 850^{o}C for 500h	SQ-AG2
5	Solution treated at 1175^{o}C for 40 minutes, water quenched and cold rolled (40% reduction)	SQ-CW

2.5 Low Cycle Fatigue Behaviour

2.5.1 Low Cycle Fatigue Testing

For studying LCF behaviour, cylindrical LCF specimens with gauge section of 5.5 mm diameter, 15 mm length and threaded ends of M12 x 35 mm length (on either side), and radii of curvature of 25 mm were machined from annealed material. Fig. 2.2 shows the dimensions of the cylindrical specimen used for the low cycle fatigue testing. The specimens were finished by fine machining and polishing to an average roughness of 0.5μm. Strain controlled LCF tests were conducted under fully reversed triangular waveform in total strain control mode using servo hydraulic testing system (MTS model 810, with flex test 40 controller). The triangular waveform used for all the strain controlled tests is given in Fig. 2.3. LCF tests were performed at three different temperatures namely, RT, 750°C and 850 °C using strain amplitudes ranging from ±0.20% to ±0.50% at a constant strain rate of 5×10^{-3} s^{-1}. The test matrix to study the effect of temperature on low cycle fatigue behaviour is shown in Fig. 2.4. The test frequency (*f*) at a certain total strain amplitude could be calculated by the following formula, $f = \dot{\varepsilon}/4\varepsilon_t$, where $\dot{\varepsilon}$ is the strain rate and ε_t is the total strain amplitude. LCF testing was conducted in accordance with ASTM standard E606. For carrying out high temperature testing, a three zone resistance heating furnace was used for heating the samples. The experimental set up for the high temperature fatigue testing with furnace is shown Fig 2.4a. 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. 2.4b. The temperature gradient along the gauge section was controlled within ±2°C of the required temperature.

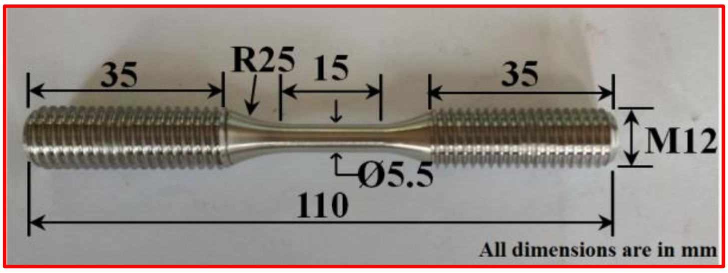

Fig. 2.2: Schematic diagram of the cylindrical fatigue specimen.

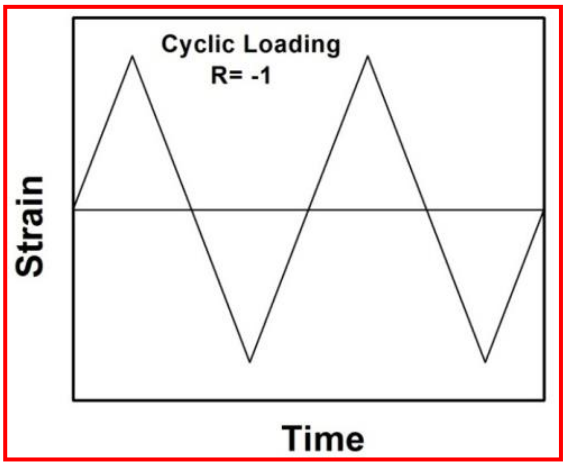

Fig. 2.3: Triangular wave form used for strain controlled fatigue testing.

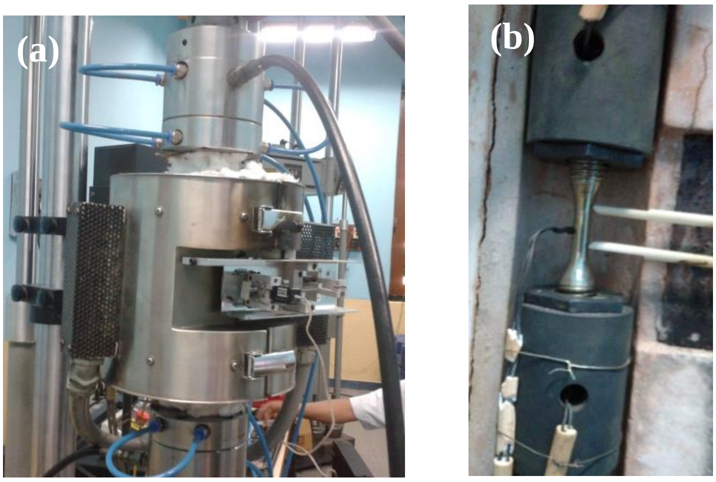

Fig. 2.4: (a) Machine set up for low cycle fatigue testing (Model: MTS 810) (b) High temperature extensometer and thermocouples mounted on the sample.

Table 2.4: Test Matrix for Low Cycle Fatigue Testing.

Test Temperature (^oC)	Strain Amplitude	Strain Rate (s^{-1})
Room Temperature	±0.20%, ±0.25%, ±0.375% ±0.42% and 0.50%	$5x10^{-3}$
750	±0.20%, ±0.25%, ±0.375% and ±0.50%	
850	±0.20%, ±0.25%, ±0.375% and ±0.50%	

2.5.2 Oxidation and Hot Corrosion

For assessing the effect of hot corrosion on LCF behaviour of Inconel 617 alloy, disc samples of 3mm thickness and 10mm diameter and fatigue samples with the required dimensions were prepared from solution annealed material. Both disc samples as well as fatigue samples were mechanically polished using emery papers of 800, 1500 and 2500 grit sizes, subsequently cloth polishing was done using alumina powder suspension. Ultrasonic cleaning was done with acetone for 10 minutes. Samples were preheated at 150^{o}C in an oven for 8 hours to remove moisture. These preheated samples were kept in furnace at 850^{o}C for 50 h, 100h, 250 h (100 x 2 cycles + 50h), 500h (100h x 5 cycles) and 1000h (100h x 10 cycles) for studying oxidation behaviour. Fig. 2.5

shows digital photographs of the disc samples after mechanical polishing (Fig. 2.5a), after oxidation for 500h (Fig. 2.5b) and fatigue sample after oxidation for 500h (Fig. 2.5c).

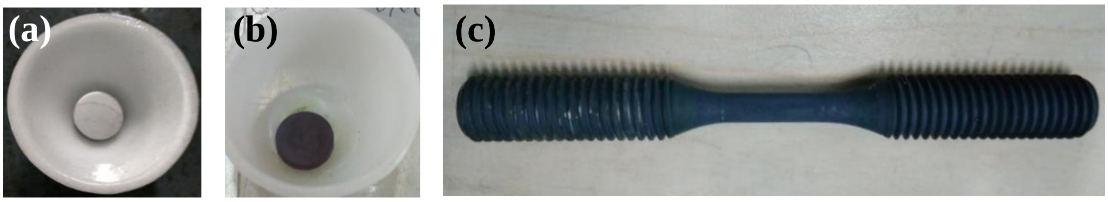

Fig. 2.5: Digital photographs of different samples: (a) disc sample as polished, (b) disc sample oxidized at 850^{o}C for 500h and (c) LCF sample oxidized at 850^{o}C for 500h.

For hot corrosion studies, both disc as well as fatigue samples were coated with super saturated aqueous solution of 90%Na_2SO_4 + 10%NaCl salts. The experimental set up for the salt spray coating is shown in Fig. 2.6, with the sample mounted with aluminium grips to avoid salt deposition on the threaded portion of the fatigue samples and to ensure the deposition of the coating only on the gauge portion of the sample. The samples were preheated to 180^{o}C-200^{o}C then sprayed with salt solution to form coating. The fatigue specimens were placed 30mm above a heating plate and rotated at 5 rpm using a dc motor while heating and spraying the salt solution. The water in the solution evaporated and the adherent salt coating was formed on the sample. Coating weight of 3-5 mg/cm^2 was maintained for both disc as well as fatigue samples. The samples were kept in oven for 8h to remove moisture after spraying. Then samples were kept in furnace for 50h, 100h, 250h (100x2 cycles + 50h), 500h(100hx5 cycles) at 850^{o}C±5^{o}C. Fig. 2.7 shows digital photographs of the disc (Fig. 2.7a) and disc sample after salt coating and pre-exposure for 500h (Fig. 2.7b) and fatigue samples after salt coating (Fig.2.7c).

LCF tests were conducted under total strain controlled mode at 850^{o}C. All the tests were conducted at total strain amplitude ($\Delta\varepsilon_t/2$) of ±0.25%, at constant strain rate

($\dot{\varepsilon}$) of 5x10^{-3} s^{-1}. XRD analysis was carried out using Rigaku 200KVa with Cu-Kα radiation for the disc samples. Longitudinal cross sections of the fatigue tested samples (near to the fracture ends), were examined with the optical microscopy to find out the depth of corrosion layer and with TEM for detailed microstructural characterization. Electron Probe Micro Analysis (EPMA) was carried out for the same samples using CAMECA SXFive instrument (voltage: 15 kV, current: 40 nA and Source: LaB_6).

Fig. 2.6: Experimental set up for salt spray coating.

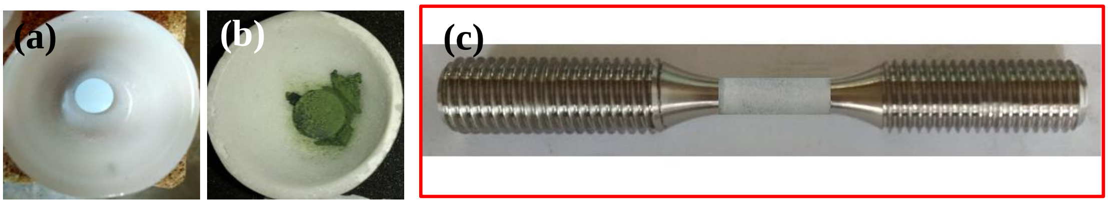

Fig. 2.7: Digital photographs of different samples: (a) disc sample as polished, (b) disc sample salt coated and pre-exposed to 850^{o}C for 500h and (c) LCF sample after salt coating.

2.5.3 Thermal Barrier Coating on Fatigue Samples

For studying the effect of TBC coating on LCF behaviour of Inconel 617 alloy, thermal barrier coating of Yittria (8%) stabilised Zirconia (YSZ) was deposited on the fatigue samples prepared from solution annealed material, using atmospheric plasma spray (APS) coating process. The atmospheric plasma spray coating process was performed by M/s. Spraymet Surface Technologies Pvt. Ltd., Bangalore, India. Fig. 2.8 shows the cross section of plasma spray gun with details of the process. Fatigue samples were initially grit blasted using alumina powder (Al_2O_3) and then were deposited with a bond coat of NiCrAlY (Metco Amdry962, Ni-22Cr-10Al-1Y, wt. %) of thickness 25-50μm. The top coat was formed on the bond coat using powdered Zirconia (ZrO_2) containing 8wt. % of Yttria -YSZ (METCO 204 C-NS, Sulzer Metco Holding AG, particle size of 20–40μm). The deposited top coat thickness on the sample was 50-75μm. The samples were rotated at uniform speed to ensure uniform coating thickness during spraying of the coating. Argon was used as the main gas to feed the powder. The sample was kept at a distance of 80mm to the gun exit point. The process parameters used for the TBC coating are given in Table 2.5. Digital photograph of the fatigue sample coated with TBC is shown in Fig. 2.9. Effect of strain amplitude on the fatigue life of the TBC coated samples was studied by conducting LCF tests at strain amplitudes from ±0.20% to ±0.50% at 850^{o}C.

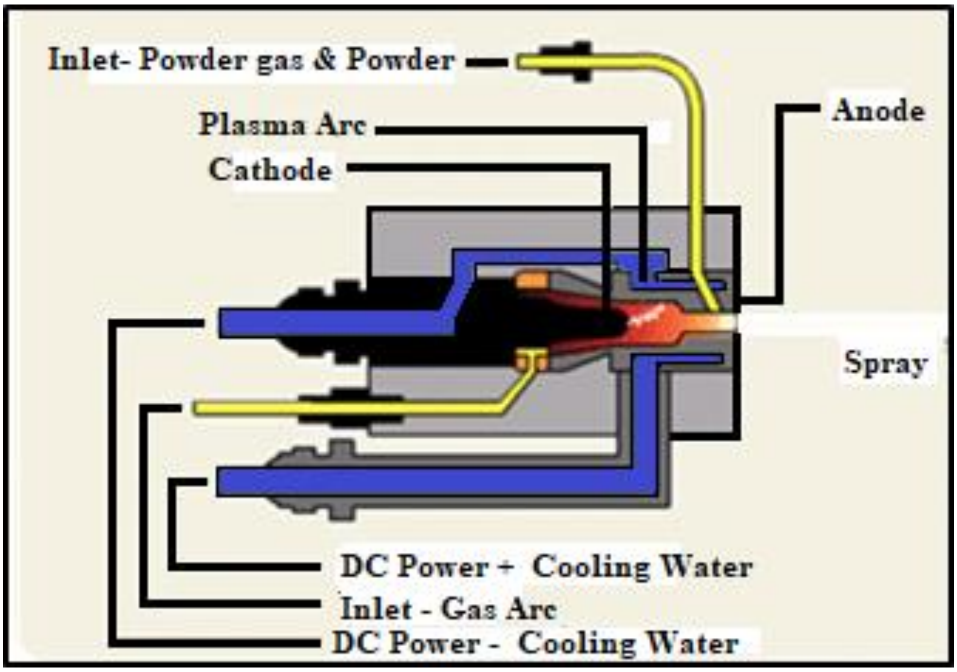

Fig. 2.8: Schematic cross section of the atmospheric plasma spray coating gun.

Table 2.5: Parameters Used for Thermal Barrier Coating.

Spraying gun	Metco 3MB
Nozzle	GH
Current (A)	500
Voltage	66-70 Volts
Primary plasma gas flow rate, Ar (slpm)	38-42
Secondary plasma gas flow rate, H_2 (slpm)	7-8
Feed rate (g min^{-1})	35- 40
Spra2ying distance (mm)	70

Fig. 2.9: Digital photograph of the LCF sample coated with thermal barrier coating.

2.6 Microstructural Characterization of the Samples after Tensile and Fatigue Testing

Fracture ends of the tensile and fatigue tested specimens were transversely sectioned, using slow speed circular saw for fractography. The fracture surfaces were cleaned in acetone using ultrasonic cleaner and were examined under scanning electron microscope operating at 30kV (Model: FEI Quanta 200F). Thin transverse slices of 0.2 mm thickness were sectioned from the region close to the fractured ends of the specimen with slow speed diamond cutter to prepare TEM foils. Similar procedure was followed for foil preparation and electro polishing of the foils as that described earlier (section 2.3). Microstructural characterization of the foils was carried out using a transmission electron microscope (Model: FEI Tecnai 20 G^2) operating at 200 kV.

2.7 Chapter Summary

1. To define DSA regime, tensile testing was carried out on Inconel 617 alloy in solution annealed condition at temperatures ranging from RT to 900^{o}C, and at three different strain rates of $5x10^{-4}$ s^{-1}, $5x10^{-3}$ s^{-1} and $1x10^{-2}$ s^{-1}.
2. Tensile testing was carried out at RT and 700^{o}C to study work hardening behaviour in different conditions of the alloy namely solution annealed, aged and cold worked conditions.
3. Low cycle fatigue tests were carried out at RT, 750^{o}C and 850^{o}C at a strain rate of $5x10^{-3}$ s^{-1}.
4. Effect of oxidation and salt environment on LCF behaviour was studied by conducting LCF tests at 850^{o}C on samples exposed at 850^{o}C to various durations with and without coating with NaCl (10%) + Na_2SO_4 (90%) salt.
5. Effect of TBC on LCF behaviour was studied by conducting LCF tests at 850^{o}C and at strain amplitudes ranging from ±0.2% to ±0.5%.