CHAPTER 2

MATERIAL AND EXPERIMENTAL METHODS

2.1 INTRODUCTION

This chapter presents the details of the test material Ti–6Al–4V and the procedure of its characterization before and after the ultrasonic shot peening (USSP), testing for corrosion and low cycle fatigue. The procedures of characterization using different types of equipment like optical microscope, scanning electron microscope, transmission electron microscope, electron probe micro analyzer, X-ray diffractometer are described. The procedures of the evaluation of surface roughness and microhardness are also given. The test matrix and procedure for electrochemical corrosion, hot corrosion, tensile tests, and low cycle fatigue tests are presented.

2.2 MATERIAL

Alloy Ti–6Al–4V was procured from M/s Mishra Dhatu Nigam Limited, Hyderabad, in hot rolled and annealed condition in the form of rods of 50 mm diameter. Pieces of 12 mm diameter were solution treated (ST) in the α+β phase field (Fig. 1.7) at 950 °C for 1h in argon atmosphere and cooled to room temperature. Its chemical composition is given in Table 2.1.

Table 2.1 Chemical composition of the alloy Ti–6Al–4V.

Elements	Ti	Al	V	C	O	N	H	Fe
Amount (wt. %)	Balance	6.24	4.11	0.015	0.163	0.004	0.005	0.040

2.3 ULTRASONIC SHOT PEENING (USSP) TREATMENT

The equipment used for USSP is shown in Fig 2.1. The ultrasonic shot peening system comprises of an auditory assembly with piezoelectric transducer, booster and sonotrode.

Fig. 2.1 The peening head (left) and the central unit (right) of the ultrasonic shot peening device.

Acoustic assembly generates mechanical vibration and transfers it to hard balls of 100C6 grade steel to put them in rapid motion. Ultrasonic waves with 20 kHz frequency are emitted by the piezoelectric transducer and amplified. Peak to peak vibration amplitude remained constant (80 μm) during the USSP. Because of the high vibrational frequency of the system, the sample surface was impacted continually by a large number of steel balls within a short time and surface of the sample was plastically deformed at high strain rate. USSP treatment was given to disc shaped small pieces of 5 mm thickness and 12 mm diameter, sectioned from the solution treated blank of 12 mm diameter using StressVoyager (SONATS) as per the processing parameters

presented in Table 2.2, for subsequent characterization of their microstructure, corrosion resistance and other mechanical properties.

Table 2.2 Processing parameters for ultrasonic shot peening.

Ultrasonic frequency (kHz)	**Vibration amplitude (μm)**	**Ball diameter (mm)**	**Processing duration (minute)**
20	80	3	0.25
			0.5
			1.0
			2.5
			5.0
			7.5
			10.0
			15.0
			30.0

2.4 MICROSTRUCTURAL CHARACTERIZATION

The samples were characterized by optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM) and X-ray diffractometer (XRD) both in the non-USSPed and USSPed conditions.

2.4.1 Optical Metallography

Specimens for optical metallography were prepared from the non-USSPed and USSPed pieces. Samples for optical metallography were mechanically polished on emery paper from 1/0 to 4/0. Final polishing was carried out on cloth, mounted on a smooth rotating polishing wheel, using suspension of alumina powder in water as abrasive. The polished samples were etched with a solution of 10%HF+5%HNO_3+ 85%H_2O (volume percent) at room temperature and the microstructures were examined using Metalux-3 optical microscope at different magnifications.

2.4.2 Scanning Electron Microscopy (SEM)

The surface morphology of USSPed as well non-USSPed samples was examined, using SEM (FESEM Quanta 200 FEG) at 30 kV. The USSPed samples were sectioned perpendicular to the shot peened surface along the diameter, to examine their microstructures from the shot peened surface towards the interior. The surface morphology of corroded samples was also examined. The hot corroded specimens were washed in hot distilled water and subjected to ultrasonic cleaning in acetone to remove salt particles and were finally dried. Further, the hot corroded samples of 12 mm diameter and 5 mm thickness were sectioned in two halves and mechanically polished to examine the depth of the oxide scale formed. The products resulting from hot corrosion of the specimens were analyzed using energy dispersive spectroscopy (EDS). The distribution of different elements was analyzed using electron probe micro-analyzer (EPMA, JOEL; JXA- 8230) at 30 kV with wavelength dispersive spectroscopy (WDS).

The fracture behavior of alloy Ti–6Al–4V resulting from tensile and fatigue testing at different strain amplitudes was characterized. Before carrying out the

fractography, all the fractured ends were sectioned transversely from the gauge section of the tensile and fatigue tested samples were cleaned ultrasonically in acetone.

2.4.3 Transmission Electron Microscopy (TEM)

Phase characterization of the non-USSPed as well as USSPed samples and the determination of grain size of the USSPed region were carried out using TEM (TECNAI G^2 20) at 200 kV. Nearly 500 µm thick section of the shot peened region was sliced (parallel to shot peened surface) using a thin diamond coated circular saw. About 10 µm thick shot peened surface region was removed using emery paper to flatten the uneven shot peened surface and the thickness of the transverse slice was reduced to about 50 µm by mechanical polishing, from opposite side of the USSPed surface. Discs of 3 mm diameter were punched out from the thinned slice and TEM foils were prepared by electrolytic thinning in an electrolyte containing 6% perchloric acid, 60% methanol and 34% n-butanol (by volume), cooled to -30 °C, at 30 volt, using a twin jet polisher (FISHIONE, Model 110). For the USSPed samples TEM foils were prepared with careful mechanical polishing from the untreated side up to 70-75 µm then polishing from the treated side by 4/0 emery paper up to 50 to 55 µm, to have smooth surface on the treated side too. Electro-jet polishing was carried out for these samples to make foils.

The deformation behavior of LCF tested specimens was examined by TEM. Foils for TEM were prepared from thin transverse sections of the fatigue tested specimens from the region close to the fractured end by electrolytic thinning referred to above.

2.5 X-RAY DIFFRACTION (XRD)

X-ray diffraction (Model: D8 advance BRUKER) studies were carried out for characterization of phases, evaluation of grain size and lattice strain of the non-USSPed and USSPed samples. Cu-Kα radiation of wavelength 1.5402Å with Ni filter was used. The various oxide products formed on the non-USSPed and USSPed specimens, resulting from exposure in air, Type-1 salt, Type-2 salt mixture and Type-3 salt mixture at high temperature for 100h were characterized by XRD.

2.6 ROUGHNESS MEASUREMENT

The roughness of the USSPed as well as non-USSPed surface was determined using surface roughness measuring tester (Mitutoyo, model no. SJ410). The roughness was measured ot different regions of the sample and the average value was taken.

2.7 HARDNESS TESTING

Microhardness profile of the USSPed and non-USSPed samples from the top surface towards interior was determined on the surface resulting from sectioning of the non-USSPed and USSPed samples along their thickness (perpendicular to flat surface), using Shimadzu microhardness tester with Vickers diamond indenter at an applied load of 50 gram for dwell time of 5 second.

2.8 STRESS RELIEVING TREATMENT

Stress relieving treatment (SR) of USSPed samples was given out in argon atmosphere at 400°C for 1h, to examine the effect of the residual stresses associated with USSP, on the hardness, corrosion and LCF. Some USSPed samples were stress relived at 300°C for 1h.

2.9 POTENTIODYNAMIC POLARIZATION

One side of the flat surface of the disc shaped specimen of 5 mm thickness and 12 mm diameter of the non-USSPed specimen and the shot peened side of the USSPed specimen was subjected to potentiodynamic polarization, covering the flat surface of the other side as well as the circumferential surface of 5 mm thickness with lacquer. Corrosion study of the USSPed as well as non-USSPed samples was carried out in Ringer's solution (NaCl-9g/l, KCl-0.42g/l, $CaCl_2$-0.48g/l, $NaHCO_3$-0.2g/l), with pH of 7.2, using a GAMRY potentiostat (PC4 series) under potentiodynamic polarization. Conventional three electrodes were used; one as reference saturated calomel electrode, graphite rod as counter electrode and the test sample as working electrode. Potentiodynamic polarization curves were recorded at a scan rate of 5mV/s.

The potentiodynamic polarization was carried out in the range of -0.5 to +1.5 $V_{(saturated\ calomel\ electrode=SCE)}$ with reference to the open circuit potential. Tafel extrapolation was used to determine the corrosion parameters. Anodic and cathodic corrosion potentials were recorded as voltage vs SCE plot for various current densities at room temperature. All the potentiodynamic polarization tests were repeated three times to ensure reproducibility of the results.

2.10 HOT CORROSION

Hot corrosion study was carried out on the non-USSPed and USSPed specimens under three different salt/salt mixtures.

2.10.1 Salt Composition and Procedure of Salt Coating

Salt/mixed salt were applied on one flat side of the disc shaped specimens of the non-USSPed and shot peened side of the USSPed specimen using a spray gun.

100%NaCl (Type–1 salt), 75%Na_2SO_4+25%NaCl (Type–2 mixed salts) and 90%Na_2SO_4+5%NaCl+5%V_2O_5 (Type–3 mixed salts) were used to study salt induced corrosion behavior of the alloy Ti–6Al–4V. Samples were cleaned with acetone before salt spraying and aqueous solutions of the salt/salt mixtures referred to above were sprayed using a spray gun from a distance of about 20 cm. While spraying, the samples were exposed from the underneath to temperature of 130-170°C to remove moisture from the sprayed salt and deposit the salt. The set-up of salt coating and air brush are shown in Fig. 2.2.

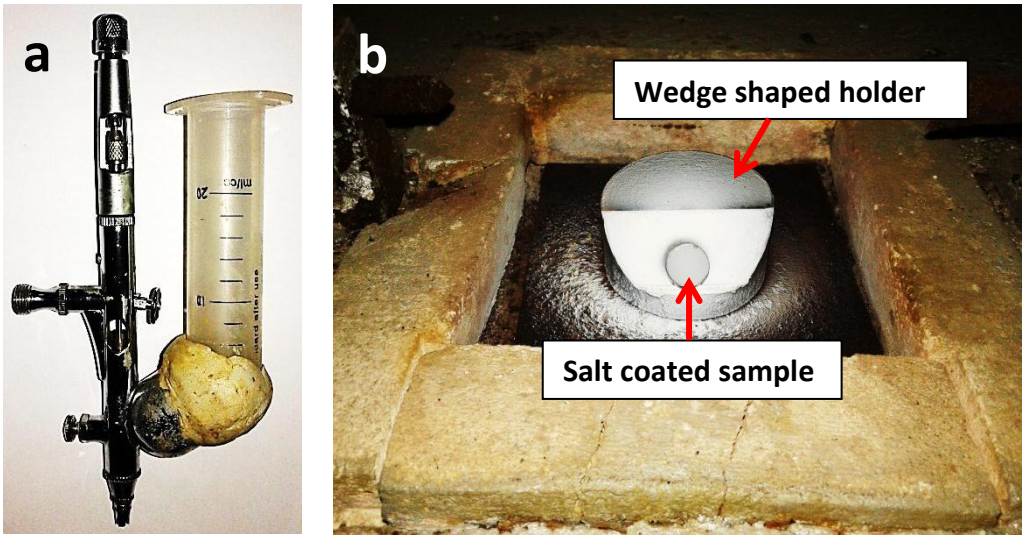

Fig. 2.2 (a) Air brush (Model-BD203) and (b) open furnace for salt coating.

An amount of 5-6.5 mg/cm^2 of NaCl salt and comparable amounts of the other two salt mixtures were deposited separately on the samples for hot corrosion study. Corrosion tests were performed keeping the as-sprayed samples in silica crucible in an electric resistance heating furnace at 400, 500 and 600 °C for a period of 100h. Specimens were subjected to heating and cooling cycles with different time periods of exposure, initially of 1h in each cycle of heating and cooling for the first 5 cycles,

subsequently of 5h in the next 4 cycles and finally of 25h for the last 3 cycles for total duration of 100h (Fig. 2.3). Non-sprayed samples were also exposed to same conditions for the purpose of comparison.

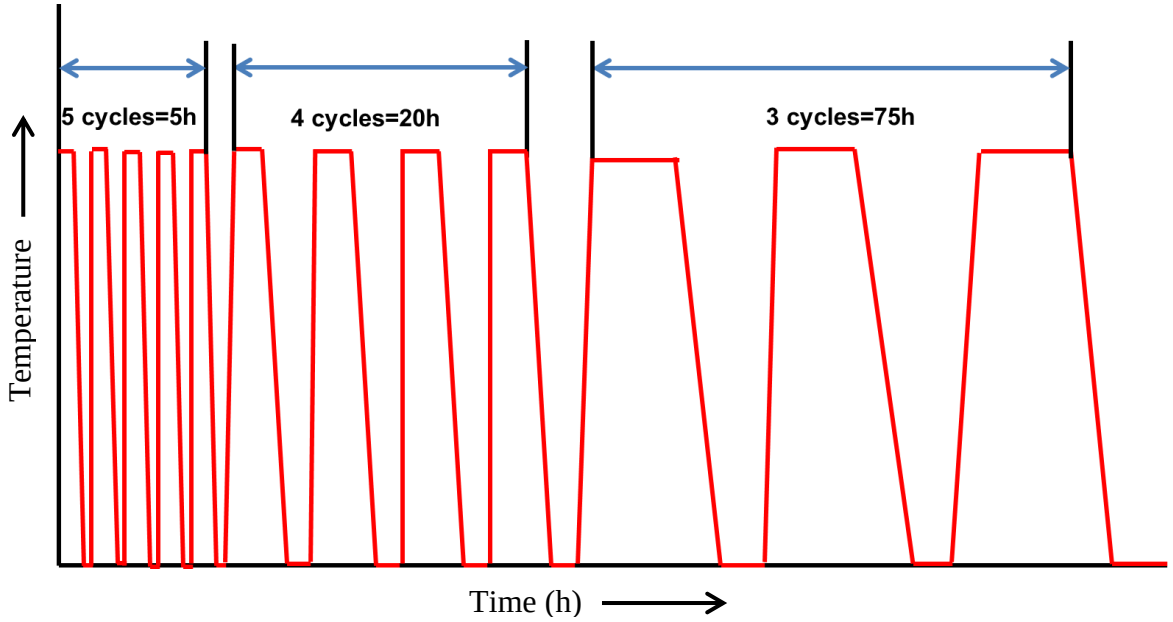

Fig. 2.3 Schematic diagram of thermal cycling during hot corrosion.

2.10.2 Weight Gain Measurement

The samples were taken out from the electric resistance heating furnace after completion of one thermal cycle, as mentioned in the experimental procedure, cooled to room temperature for about 20 minute, weighed and placed inside the furnace for the next thermal cycle. Weight gain of the non-sprayed and sprayed samples was measured along with the silica crucible after each cycle, using an electronic weighing balance of 0.1 mg sensitivity. The spalled scale was also included for the measurement of weight change to determine the rate of corrosion. The weight change of the sample was calculated using the equation 2.1.

$$\Delta W = W_f - W_i \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (2.1)$$

where ΔW is weight change, W_i is initial weight of the sample before the hot corrosion and W_f is final weight of the sample after hot corrosion. The change in weight was observed in milligram (mg). The above process was continued for each thermal cycle up to 100h.

2.11 TENSILE TESTS

Tensile tests were conducted at room temperature, at a nominal strain rate of $10^{-3}s^{-1}$ using a 100kN screw-driven Instron™ Universal Testing Machine (Model 4206). LCF specimen geometry was used for tensile testing.

2.12 LOW CYCLE FATIGUE (LCF) TESTS

LCF tests were conducted at different total strain amplitudes from ±0.60% to ±1.0%, at a constant strain rate of $5\times10^{-3}\ s^{-1}$ under fully reversed loading (R = –1) with triangular wave profile. A servo-hydraulic MTS™ fatigue testing machine (Model 810) of 50kN capacity, equipped with fully automatic Flex Text 40 controller of MTS™ (Fig. 2.4) was used for LCF testing.

Cyclic strain was controlled mounting an extensometer of 10 mm gauge length (Model: MTS 632.13C-20) on gauge section of the specimen. Cylindrical fatigue specimens were machined from the heat treated blanks, with gauge length and diameter of 15 mm and 5.5 mm respectively, shoulder radii of 25 mm and threaded ends of 30 mm length and 12 mm diameter. Gauge section was polished with emery papers of 1/0 to 4/0 grades and finally with alumina lapping powder. The polished LCF specimens were subjected to USSP with hard steel balls of 3mm diameter, at a frequency of 20 kHz for 5 minute, using StressVoyager (SONATA). The steel balls

strike the specimen surface and the specimen is kept in horizontal position and rotated along its axis at 5 rpm, to ensure uniform shot peening of the cylindrical surface. The schematic of USSP is shown in Fig. 2.5.

Fig. 2.4 Servohydraulic MTS for fatigue testing.

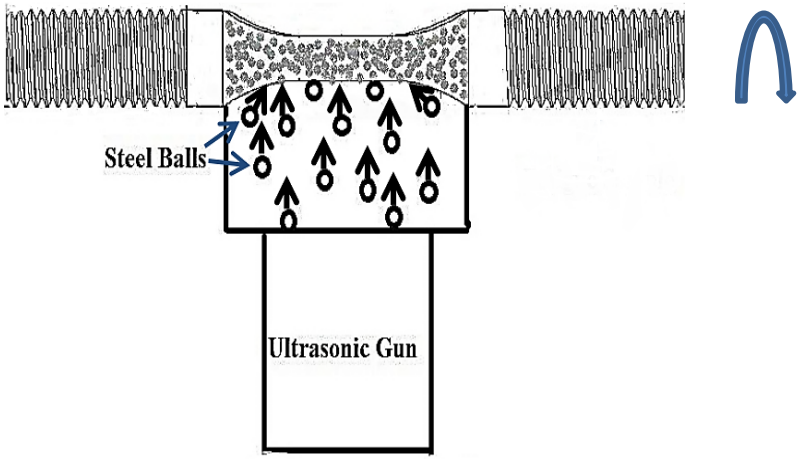

Fig. 2.5 Schematic diagram showing ultrasonic shot peening of the LCF specimen.

The test matrix of low cycle fatigue tests is shown in Table 2.3. The elastic, plastic and total strain components along with stress amplitudes, both in tensile and compressive part of each cycle, were stored and displayed by controller software.

All the fatigue tests were repeated to ensure reproducibility of the results. LCF tests were also conducted for 2.5 and 7.5 minute USSPed sample at total strain amplitude of ±0.75%.

Table 2.3 Test matrix of low cycle fatigue tests.

Conditions	**Total Strain Amplitude (%)**	**Strain Rate**
non-USSPed	±0.60, ±0.65, ±0.70, ±0.75, ±0.80, ±0.90, ±1.0	$5\times10^{-3}s^{-1}$
5 minute USSPed	±0.60, ±0.65, ±0.70, ±0.75, ±0.80, ±0.90, ±1.0	$5\times10^{-3}s^{-1}$
5 minute USSPed+SR	±0.60, ±0.65, ±0.70, ±0.75, ±0.80, ±0.90%, ±1.0	$5\times10^{-3}s^{-1}$

SR= stress relieving