CHAPTER 2

MATERIALS AND EXPERIMENTAL METHODS

2.1 INTRODUCTION

This chapter describes the material of the present investigation and experimental details related to the heat treatment, process of surface roughness generation by manual grinding on emery paper and grain refinement by ultrasonic shot peening (USP). Method of hot corrosion and high cycle fatigue (HCF) testing are also discussed. Characterization techniques like optical microscopy, scanning electron microscopy, transmission electron microscopy, electron probe micro analysis, and X-ray diffraction are described.

2.2 MATERIALS & HEAT TREATMENT

The superalloy IN718 was procured from M/s Mishra Dhatu Nigam Limited, Hyderabad, in solution annealed condition in the form of rods of 20 mm diameter. The alloy was subjected to double aging heat treatment cycle (720˚C for 8h, furnace cooled @ 55˚C/h to 620˚C, held at 620˚C for 8h followed by forced air cooling). The chemical composition of the as received material is given in Table 2.1.

Table 2.1: Chemical composition of the superalloy IN718 (wt.%).

Ni	**Cr**	**Nb+Ta**	**Ti**	**Al**
53.5	17.91	5.22	1.04	0.5
Mo	**C**	**Si**	**Mn**	**Fe**
3.10	0.03	0.03	0.02	Balance

The heat treated rods were sliced transversely to produce disc shaped samples of 2 mm thickness and 20 mm diameter for hot corrosion studies. The heat treated rods were also sectioned longitudinally to blanks of 98 and 110 mm lengths to prepare cylindrical HCF specimens of hour glass type with zero gage length and 15 mm gage length, respectively.

2.3 GENERATION OF SURFACE ROUGHNESS

The sectioned discs were mirror polished and subsequently mechanically ground on SiC emery papers of #400, #600, #800 and #1000 grit for one minute in unidirection. The surface roughness was measured by Mituyotu surface roughness tester.

2.4 ULTRASONIC SHOT PEENING (USP) TREATMENT

The equipment used for surface modification by ultrasonic shot peening (USP) is shown in Fig 2.1. The ultrasonic shot peening system comprises of an auditory assembly with piezoelectric transducer, booster and sonotrode. 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 USP. 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 the surface of the sample was plastically deformed at high strain rate. USP treatment using Stress Voyager (SONATS) was given on flat surface of disc shaped small pieces of 5 mm thickness and 20 mm diameter.

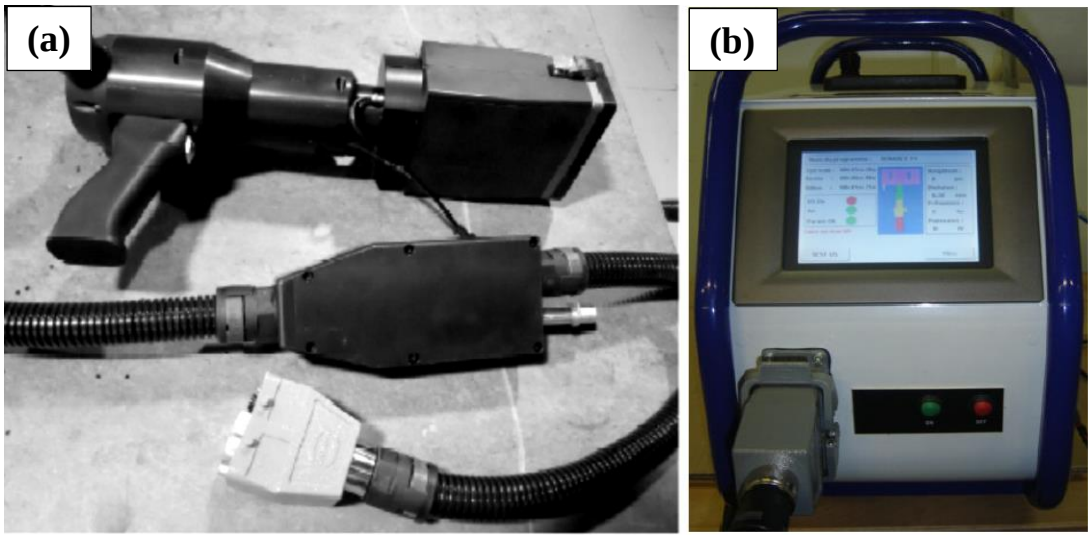


Fig. 2. 1: Components of ultrasonic shot peening device: (a) the peening head and (b) the central unit of the ultrasonic shot peening device.

2.5 HOT CORROSION

Low temperature hot corrosion study was carried out on the samples of different surface roughness and the USP treated specimens with three different salt/salt mixtures. The details are given below:

2.5.1 SALT COATING ON FLAT AND CYLINDRICAL SPECIMENS

Three different salt coatings of 100% NaCl (1S salt), 60% Na_2SO_4 + 40% V_2O_5 (2SM salt mixture), and 75% Na_2SO_4 + 15% NaCl + 10% V_2O_5 (3SM salt mixture) were used to study hot corrosion behavior of the superalloy IN718. 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. The samples were preheated to temperature of 150-200°C for 10 minutes before salt coating to remove moisture from the sprayed salt solution. The set-up of salt coating and air brush are shown

in Fig. 2.2. The specimens were weighed before and after the salt deposition, and weight of the coating per unit area was determined.

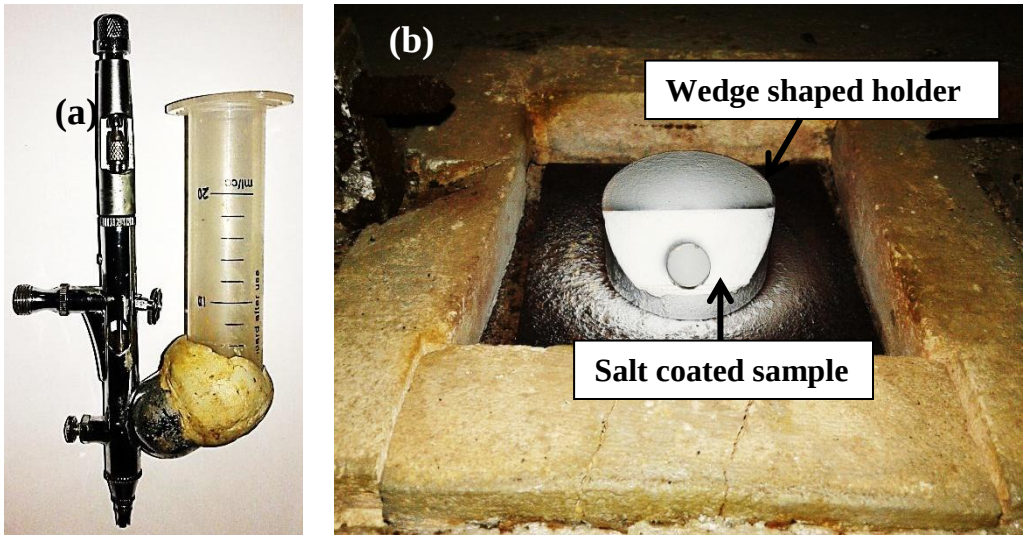


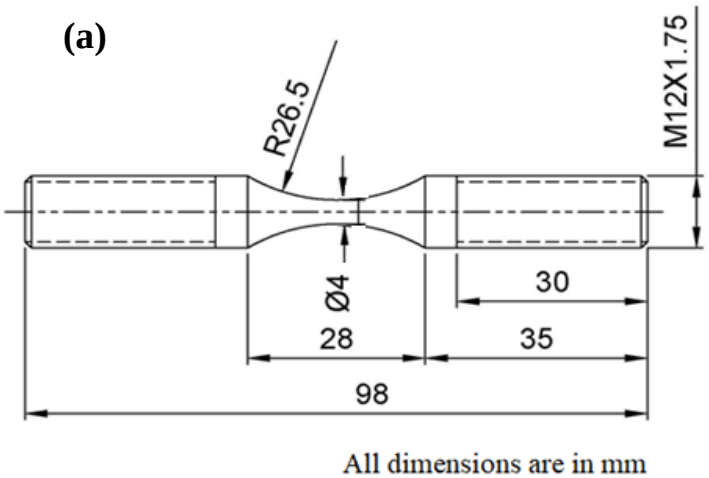
Fig. 2. 2: Salt coating set-up: (a) Air brush (Model-BD203) and (b) hot plate for salt coating.

An amount of 3.5-4 mg/cm^2 of NaCl salt and the other two salt mixtures was deposited separately on the each sample for hot corrosion study. Hot corrosion tests were performed keeping the as-sprayed samples in silica crucible in an electric resistance heating furnace at 600 and 700°C with temperature control of ±2ºC using the Sandvik digital controller for a period of 100h. The specimens were subjected to heating and cooling cycles with different time periods of exposure, initially of 0.5h followed by 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 the total duration of 100h. Bare samples were also exposed to similar conditions for the purpose of comparison. The weight of silica crucibles with test specimen was measured at the end of each cycle using

an electric balance (Mettler Toledo) with sensitivity of 0.1 mg. The weight of the spalled scale was also taken into account.

2.5.2 PRE-HOT CORROSION OF HCF SPECIMENS

The salt mixture of 75% Na_2SO_4 + 15% NaCl + 10% V_2O_5 (3SM salt mixture) was used for pre hot corrosion of the high cycle fatigue specimens. The geometry of the hour glass type HCF specimen is shown in Fig. 2.3 a. Fatigue samples were cleaned with acetone, weighed, and preheated to temperature of 150–200°C by holding the sample horizontally at a height of ~ 30 mm above the hot plate and rotating at 5 rpm (Fig. 2.3 b). Salt spraying was carried out in middle section of the hour glass sample. Salt deposit of 3.5 - 4 mg/cm^2 was found to form a uniform and intact salt layer on the HCF specimen. Salt coated HCF specimens were subjected to high cycle fatigue test at the frequency of 30 Hz. It was observed that the salt layer peeled off soon after the start of the fatigue test. Therefore, the salt coated samples were hot corroded at 600^{o}C±2^{o}C for 100 h prior to the HCF test at 600^{o}C±1^{o}C in air. The hot corroded samples were kept in an oven at 150^{o}C±2^{o}C to avoid absorption of moisture from air before HCF test.



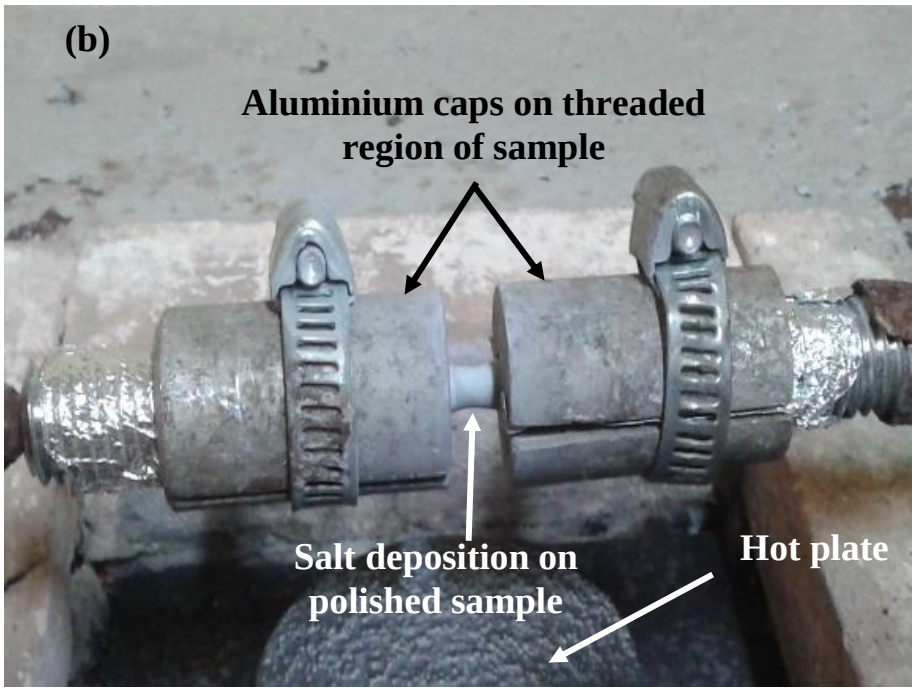


Fig. 2. 3: (a) Hour glass geometry of HCF test specimen for R=-1, (b) Digital photographs of salt deposition system in central region of the curved section.

2.6 HIGH CYCLE FATIGUE (HCF) TESTS

Cylindrical HCF specimens with gage section of 15 mm length and 5.5 mm diameter were used to study the effect of mean stress on high cycle fatigue (Fig. 2.4). The curved section and gage section of the HCF specimens as shown in Fig. 2.3 (a) and Fig. 2.4 were mechanically polished by 3/0 and 4/0 grades of emery papers, followed by alumina powder to remove machining marks, if any, and make the surface smooth. HCF specimens were washed with distilled water and subsequently cleaned with acetone. HCF tests were performed at stress ratios of -1, 0.5 and 0.7 at 600^{o}C±1^{o}C at a constant frequency of 30 Hz.

HCF tests were performed on a fully computer-controlled Servo Hydraulic MTS Testing Machine (Model-810) (Fig. 2.5) of 50 kN capacity with Flex Test 40 digital

controller interface equipped with a split electric resistance heating furnace (Model-652.01D, Sr. No. 0114704) with temperature control of ±1°C accuracy. High cycle fatigue tests under symmetrical loading (R = -1), were conducted using hour glass specimen geometry (Fig. 2.3 a). Fully reversed ($R = -1$) HCF tests were conducted in air under stress control mode on both the as heat treated as well as pre hot corroded specimens. Tests were carried out at 600°C±1°C, at different stress amplitudes of ±450 MPa, ±500 MPa, ±550 MPa, ±600 MPa, ±650 MPa, ±700 MPa and ±750 MPa at a constant frequency of 30 Hz.

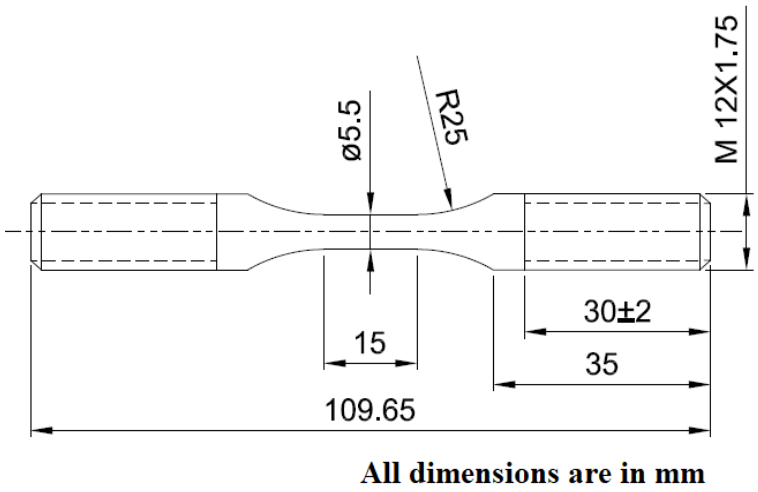


Fig. 2. 4: Geometry of HCF test specimen used at stress ratio of 0.5 and 0.7

Fig. 2. 5: 50 kN Servo material testing system.

2.7 DIGITAL PHOTOGRAPHY

The hot corroded specimens were visually examined after every heating cycle. The features of corrosion scale were recorded using a digital camera (Sony-Cybershot).

2.8 MICROSTRUCTURAL CHARACTERIZATION

The samples of the superalloy IN718 were characterized by optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD) and EPMA both in the non-USP and USP treated conditions.

2.8.1 OPTICAL METALLOGRAPHY

Samples for optical metallography were mechanically polished on emery paper from 1/0 to 4/0 grade. Final polishing was carried out on sylvetcloth, mounted on a smooth rotating polishing wheel, using suspension of alumina powder in water as

abrasive. The polished samples were etched with Kallings reagent No.2 (5g $CuCl_2$, 100 ml HCl and 100 ml ethanol) at room temperature and the microstructures were examined using Leitz Metalux-3 optical microscope at different magnifications.

2.8.2 SCANNING ELECTRON MICROSCOPY (SEM)

SEM (FESEM Quanta 200 FEG and Zeiss EVO/18) study with EDS spectrum of the specimens, subjected to various experimental conditions, was carried out to examine the surface, corrosion scale and cross-section. The hot corroded specimens were washed in hot distilled water and subjected to ultrasonic cleaning in acetone to remove loose salt particles and were finally dried. Further, the hot corroded samples of 20 mm diameter and 5 mm thickness were sectioned in two halves along the diameter, perpendicular to treated surface and the resulting section was mechanically polished to examine the depth of the oxide scale formed on the surface. 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, CAMECA SXFive) at 15 kV with wavelength dispersive spectroscopy (WDS).

The fracture behavior of the superalloy IN718 resulting from high cycle fatigue testing at different stress amplitudes was characterized using SEM. Before carrying out the fractography, the fractured ends of 3-4 mm length were sectioned transversely from the fractured fatigue tested samples and cleaned ultrasonically in acetone.

2.8.3 ELECTRON PROBE MICRO ANALYSIS (EPMA)

The cross sections of the hot corroded samples were examined for elemental distribution under Electron Probe Micro Analyzer (EPMA) CAMECA SX Five

instrument at a voltage of 15 kV and current of 10 & 20 nA with a LaB6 source in the electron gun.

2.8.4 TRANSMISSION ELECTRON MICROSCOPY (TEM)

Phase characterization of the non-USP as well as the USP treated samples and the determination of grain size of the USP affected region was carried out using TEM (TECNAI 20 G^2) at 200 kV. Nearly 500 µm thick section of the shot peened region was sliced, parallel to the shot peened surface, using a thin diamond coated circular saw. About 10 µm thick region of the shot peened surface as 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 the opposite side of the USP treated 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 (STRUAERS). For the USP treated 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 TEM foils.

2.9 X-RAY DIFFRACTION (XRD)

X-ray diffraction studies were carried out using RIGAKU X-ray diffractometer for characterization of phases, evaluation of grain size of the non-USP and USP treated samples. Cu-Kα radiation of wavelength 1.5402Å with Ni filter was used. The various

oxide products formed on the non-USP and USP treated specimens, resulting from exposure in air, 1S salt, 2SM salt mixture and 3SM salt mixture were analyzed by XRD.

2.10 SURFACE ROUGHNESS MEASUREMENT

The roughness of the surfaces ground with emery papers of different grits and also the USP treated surfaces was determined using surface roughness measuring tester (Mitutoyo, Model no. SJ410). The roughness was measured at different regions of the sample and the average value was taken and (R_a) was estimated to be 0.46, 0.28, 0.16 0.09, 1.43 µm respectively for the samples ground on 400, 600, 800, 1000 grit and 5 min USP treated.