CHAPTER 2

MATERIAL AND EXPERIMENTAL METHODS

2.1 Introduction

This chapter presents the details of the test material AA7075 aluminium alloy and its heat treatment used in the present investigation. The process of ultrasonic shot peening (USSP) and characterization of the material following USSP is described. The processes of characterization using different techniques like optical microscopy, scanning electron microscopy, transmission electron microscopy, atomic force microscopy, X-ray diffraction are described. Test details like geometry and fabrication of specimen for mechanical testing and corrosion test are also included.

2.2 Material

The AA7075 aluminium alloy was procured from M/s Hindalco Industries Limited, Renukot, India, in the form of cylindrical bar of 54 mm diameter and 1000 mm length. The nominal composition, determined by spark emission spectroscopy is recorded in Table 2.1.

The material was subjected to retrogression and re-aging treatment which involoved

TABLE 2.1: Chemical composition of the AA7075 Al alloy (wt.%).

Zn	Mg	Cu	Si	Cr	Mn	Fe	Al
4.89	2.12	1.52	0.33	0.21	0.09	0.007	Balance

solution treatment at 470°C for 30 min, pre-aging at 120°C for 24 h, followed by retrogression at 200°C for 10 min, quenching, and subsequent secondary aging at 120°C for 24 h. The schematic of heat treatment cycle is shown in Fig. 2.1

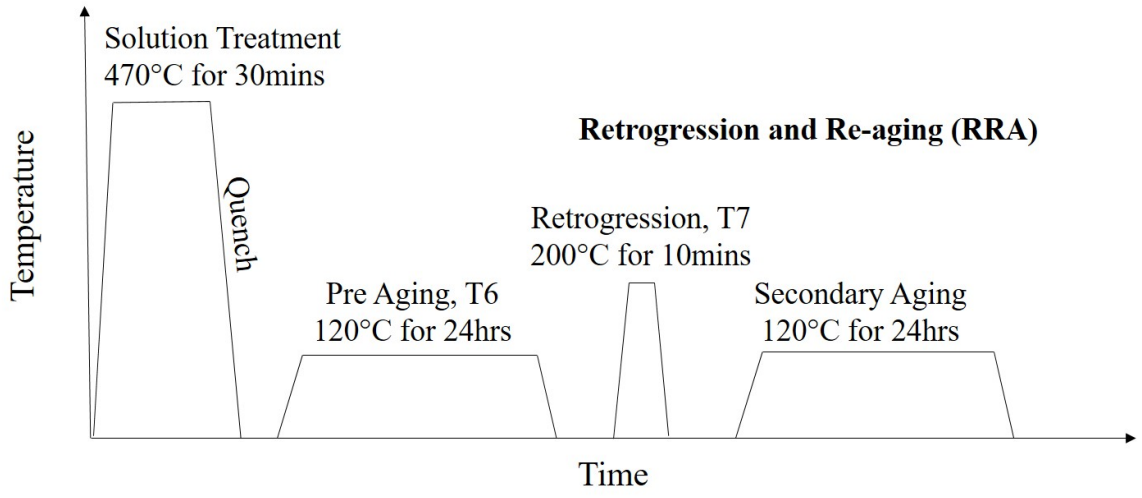

FIGURE 2.1: Schematic of the retrogression and reaging heat treatment of AA7075.

2.3 Ultrasonic Shot Peening (USSP) Treatment

The equipment used for the USSP is shown in Fig. 2.2. 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.

Vibration amplitude remains constant at 80 μm during the USSP. When the balls are resonated, the surface of the sample to be treated is impacted by a large number of flying balls over a short period of time. The impact directions of the balls onto the sample

FIGURE 2.2: The peening head (left) and the central unit (right) of the ultrasonic shot peening device.

TABLE 2.2: Processing parameters for ultrasonic shot peening.

Ultrasonic frequency (kHz)	**Vibration amplitude (μm)**	**Ball diameter (mm)**	**Processing duration (seconds)**
20	80	3	15
20	80	3	30
20	80	3	60
20	80	3	180
20	80	3	300

surface are random and surface of the sample is plastically deformed at high strain rate. USSP treatment was given to disc shaped small pieces of 4 mm thickness and 5 mm diameter, using Stress Voyager (SONATS) as per the processing parameters presented in the Table 2.2, for subsequent characterization of their microstructure, corrosion resistance, hardness and low cycle fatigue properties.

2.4 Microstructural Characterization

The microstructures of the un-USSP and USSP treated samples were characterized by optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy

(TEM). Phase stability following USSP was assessed by X-ray diffraction.

2.4.1 Optical Microscopy

Specimens for optical microscopy were prepared from the un-USSP treated and USSP treated pieces. Samples for optical metallography were mechanically polished on emery papers from 1/0 to 4/0. Final polishing was carried out on cloth, mounted on a smooth rotating polishing wheel, using diamond suspension (1 μm) and finally with colloidal silica (0.05 μm, $\sim$ 8.5 pH). The polished samples were etched by Keller's reagent (2 ml HF(48%) + 3 ml HCl + 5ml HNO_3 + 190 ml H_2O) 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 the un-USSP as well USSP treated samples was examined, using SEM (FESEM Quanta 200 FEG) and Zeiss (EVO18) at 30 kV. The USSP treated samples were sectioned perpendicular to the shot peened surface along their diameter to examine their microstructures from the shot peened surface towards the interior. The surface morphology of corroded samples was also examined. The products resulting from corrosion were analyzed using energy dispersive spectroscopy (EDS).

Fracture morphology of the alloy AA7075 resulting from fatigue testing at different strain amplitudes was characterized. Before carrying out the fractography, small pieces of $\sim$ 4 mm length were sectioned transversely from the fractured end of the tested samples

and cleaned ultrasonically in acetone for 5 min. The corrosion tested specimens were subjected to ultrasonic cleaning twice to remove the deposits resulting from the corrosive solution.

2.4.3 Transmission Electron Microscopy (TEM)

A TECNAI 20 G^2 transmission electron microscope operating at 200 kV, equipped with a high-angle annular dark field (HAADF) detector was used to characterize the microstructure of the surface region. TEM foils of the USSP treated surface region were prepared, sectioning thin slices from the USSP treated region using a slow speed precision cutter. These slices were thinned down by mechanical polishing from the side opposite to the treated surface up to a thickness of $\sim$ 50 μm. Discs of 3 mm diameter were punched out from the thinned slice and TEM foils were prepared by electrolytic thinning from the side opposite to the treated surface, in the electrolyte containing 20% nitric acid in methanol, at -30°C, at 20 V, using a twin jet polisher (TenuPol-5).

2.4.4 X-Ray Diffraction (XRD)

X-ray diffraction of the differently treated samples was carried out by Rigaku X-ray diffractometer with Cu Kα radiation in 2θ range from 30°to 90°for characterization of phases, evaluation of grain size, lattice strain, lattice parameter and dislocation density of the un-USSP and USSP treated samples. X-ray diffraction was used also to characterize the type of corrosion products formed over the sample surface after its prolonged immersion in corrosive solution.

2.5 Mechanical Testing

2.5.1 Hardness testing

Microhardness profile of the USSP treated and un-USSP samples from the top surface towards interior was determined by sectioning the un-USSP and USSP treated samples along their thickness (perpendicular to flat surface), using Leco microhardness tester (LM248AT) with Vickers diamond indenter at an applied load of 50 g for dwell time of 10 s.

2.5.2 Tensile testing

Room temperature tensile tests were performed using a 100 kN (Instron 5982) Universal testing machine at a strain rate of 5x10^{-3} s^{-1}. Tensile specimens of dog-bone shape with gage section of 15 mm x 5 mm x 2 mm were used. Schematic of tensile test specimen is shown in Fig. 2.3.

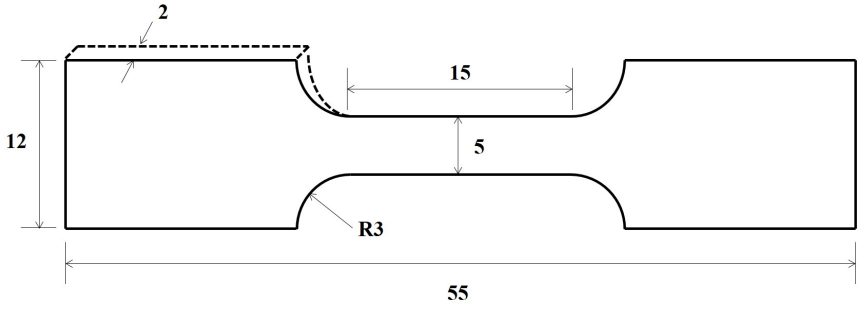

FIGURE 2.3: Geometry of the flat tensile specimen with dimensions in mm.

2.5.3 Low Cycle Fatigue (LCF) testing

Cylindrical LCF specimens were machined from the peak aged blanks, with gage 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 (Fig. 2.4).

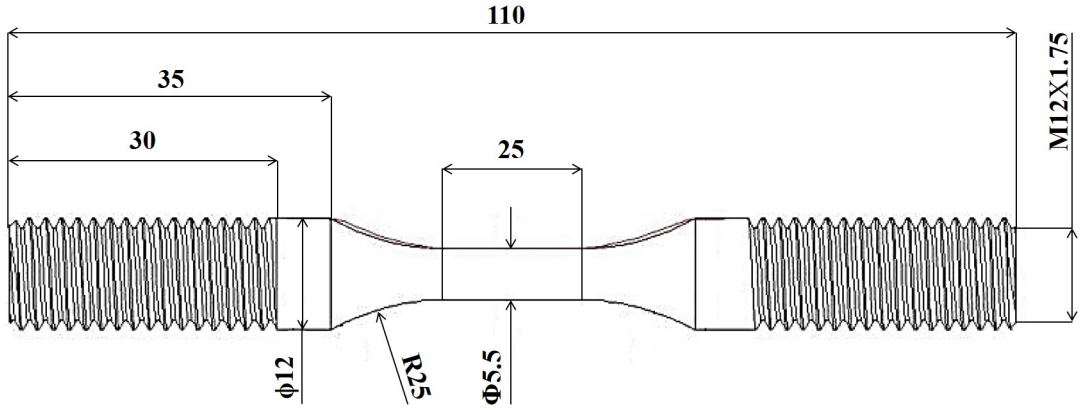

FIGURE 2.4: Schematic of the cylindrical LCF specimen with dimensions in mm.

LCF tests were conducted at different total strain amplitudes from ±0.38% to ±0.06%, at a constant strain rate of 5×10^{-3} s^{-1} under fully reversed loading (R = -1) with triangular wave profile. A servo-hydraulic MTS™ fatigue testing machine (Model 810) of 50 kN capacity, equipped with fully automatic Flex Text 40 controller of MTS™ was used for LCF testing. Cyclic strain was controlled mounting an extensometer of 10 mm gage length (Model: MTS 632.13C-20) over the gage section of the specimen. Gage 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 3 mm diameter, at a frequency of 20 kHz for different durations of treatment, using Stress Voyager (SONATS). The steel balls strike the specimen surface and the specimen is kept

TABLE 2.3: Test matrix of low cycle fatigue tests.

Treatment condition	**Strain amplitude (μm)**	**Strain rate (s^{-1})**
un-USSP USSP 30 USSP 60 USSP 180 USSP 300	±0.38%, ±0.40%, ±0.45%, ±0.50%, ±0.55%, ±0.60%	5 x 10^{-3}

in horizontal position and rotated along its axis at 5 rpm, to ensure uniform shot peening of the cylindrical surface.

The test matrix of the 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.

2.5.4 Slow Strain Rate Tensile (SSRT) testing

To evaluate the stress corrosion behavior of the alloy, the samples were tested using slow strain rate tensile testing machine in air as well as 3.5 wt.% NaCl solution. SSRT tests were performed using a 100 kN (Instron 5982) universal testing machine at a very low strain rate of 1x10^{-6} s^{-1}. Flat sheets of 2 mm thickness were USSP treated and then machined in order to prepare flat samples with dimension shown in Fig. 2.5.

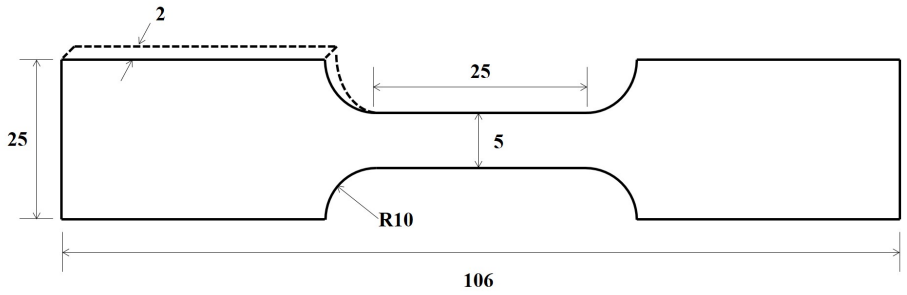

FIGURE 2.5: Dimensions of the specimen used in the SSRT test with dimension in mm.

2.6 Corrosion Measurements

2.6.1 Electrochemical measurements

Potentiodynamic Polarization (PD) and electrochemical impedance spectroscopy (EIS) were carried out using GAMRY^{TM} Potentiostat (Series: PC-4) in 3.5 wt.% NaCl solution at 27°C (±2°C). A three-electrode cell consisting of saturated calomel electrode (SCE) as reference electrode, graphite as counter electrode and the specimen as working electrode was used. The working electrode surface area was 1 cm^2 for all the specimens. The samples were exposed in 3.5 wt.% NaCl solution for 30 min to stabilise their open circuit potential, prior to potentiodynamic (PD) and electrochemical impedance spectroscopy (EIS) measurements. The test electrolyte (3.5 wt.% NaCl aqueous solution) was prepared, dissolving analytical grade NaCl in double distilled water. Potentiodynamic tests were carried out in the potential range from -0.5 V to 1.5 V at a scan rate of 1 mV/s. The EIS tests were performed imposing 10 mV sinusoidal voltage at open circuit potential of the test electrodes and changing the frequency between 100 kHz and 10 mHz. All the electrochemical studies were carried out in triplicate to ensure reproducibility of the data.

2.6.2 Static immersion testing

Static immersion tests were carried out using disc shaped samples of 5 mm diameter and 3 mm thickness, sectioned from the original rod of 54 mm diameter. Before the USSP treatment the specimens were mechanically polished using 1500 grit emery paper and USSP treated from both the sides. Following the USSP treatment, the samples were ultrasonically cleaned in acetone to remove dirt and any other foreign particles from the shot peened surface. Before immersion in the 3.5 wt.% NaCl solution each sample was weighed with an accuracy of 0.0001 mg. The samples were removed from the solution after the interval of 15 days, rinsed with double distilled water in order to remove salt and loose corrosion products, dried in oven at 60°C for 2 h and finally weighed. Three test samples were suspended for each condition, vertically in 250 ml of 3.5 wt% NaCl solution in separate beakers, at room temperature. The level of the solution in the beakers was checked every three days and maintained by adding double distilled water. After the exposure of 60 days, samples were removed from the 3.5 wt.% NaCl solution and cleaned as per the ASTM standard G1-03 and finally weighed. The corrosion rate was calculated from the weight loss method (ASTM G1-03) [80] after cleaning the samples, using the following equation.

$$CorrosionRate(\mu m/year) = \frac{KW}{AtD} \tag{2.1}$$

Where, K is a constant (8.76×10^4), t is the time of exposure (h), A is the area of the exposed samples (in cm^2), W is the mass loss (mg) and D is the density of aluminium alloy 7075 in g/cm^3.