Chapter 2

Experimental methods and Characterization Techniques

2.1 Alloy development

The material employed in this investigation is a commercially accessible 7075 aluminum alloy. Which is developed with the help of vacuum stir casting method (Figs.2.1 and 2.3). Vacuum stir casting is a liquid metallurgy technique used to fabricate metal matrix composites (MMCs) and advanced alloys with better homogeneity and fewer defects. The development of 7075 aluminum alloy through vacuum stir casting involves a series of carefully controlled steps to ensure compositional accuracy, defect minimization, and superior mechanical performance. Initially, the aluminum charge is melted at a temperature of 700–740 °C, which is maintained 40–80 °C above the liquidus point to promote complete melting and effective alloying. Alloying elements, introduced as master alloys of Zn, Mg (SF_6 gas was used to add Mg, because Mg is explosive), Cu, and Cr, are added gradually to the molten metal while stirring at low speeds to ensure dissolution, followed by a homogenization hold of 10–20 minutes. Once homogenized, the melt is subjected to vacuum stirring, wherein the crucible is either sealed within or transferred to a vacuum chamber. The chamber is evacuated to a vacuum level within the range of 10^−2 to 10^−1 mbar, significantly reducing dissolved gases and inclusions. Mechanical stirring is then carried out at a speed of 250–600 rpm, typically starting around 400 rpm, for a duration of 3–8 minutes. This combined action of vacuum and agitation facilitates degassing, inclusion flotation, and compositional uniformity. To avoid vortex formation and oxide entrapment, stirring is gradually slowed, and the melt is allowed to stabilize under vacuum. Following this, the melt surface may be protected with a flux before pouring. If chamber opening is required, venting is performed with dry argon to prevent reoxidation. The molten alloy is then poured at a controlled temperature of 680–720 °C into preheated steel or ceramic molds maintained at 200–350 °C, minimizing turbulence and air entrapment during filling. The chemical composition of developed alloy is outlined in Table 2.1. Developed alloy size was 50 mm × 109 mm × 250 mm (Fig.2.2), from which rectangle shaped 15×25 mm^2 and 35 mm long insets for rolling were cut.

Table 2.1: Composition of 7075 Al alloy determined by PMI method

	Si%	Cu%	Fe%	Zn%	Mg%	Mn%	Ti%	Cr%	Al%
Comp	0.15	1.59	0.22	5.45	2.41	0.12	0.05	0.21	89.54
REQD	0.00 0.04	1.2 2.0	0.00 0.5	5.10 6.10	2.10 2.90	0.00 0.30	0.00 0.20	0.18 0.28	Rem.

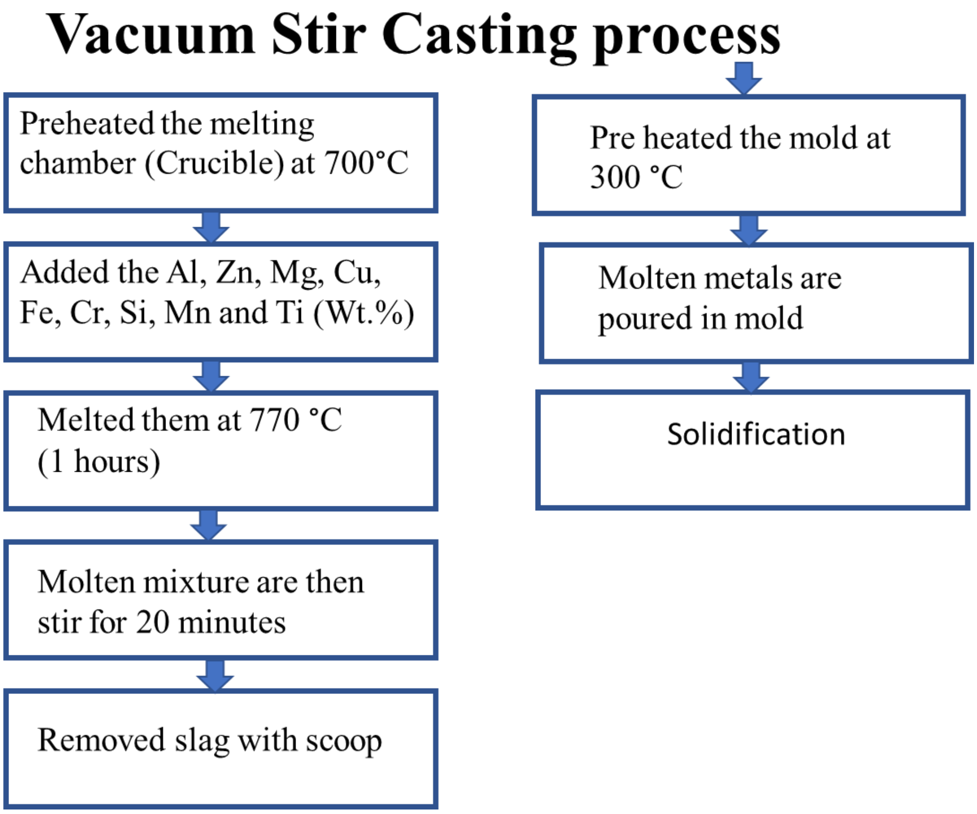

Fig. 2.1 Vacuum stir casting processing flow chart to develop 7075 Al alloy

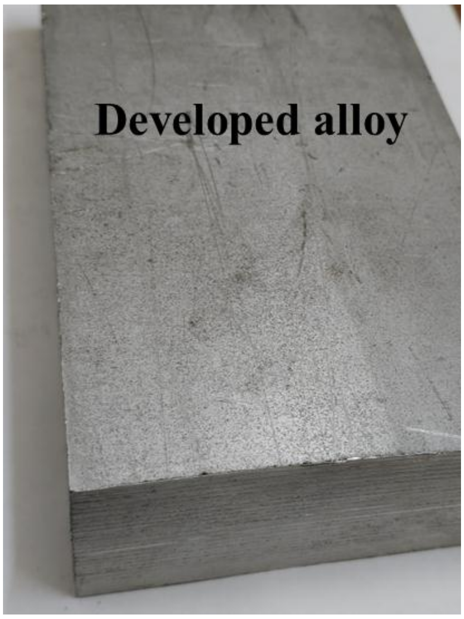

Fig.2.2 7075 Al alloy developed through vacuum stir casting

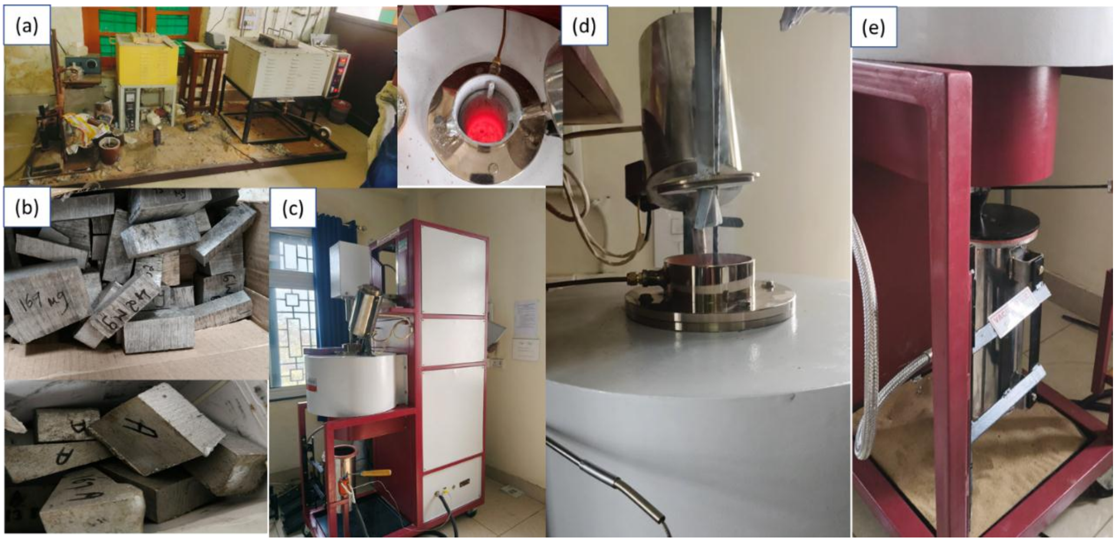

Fig.2.3 (a) Casting set up (b) Al, Mg, Zn metals, (c) Vacuum stir casting(lab), (d) Slag removal, (e) pouring of molten metal

2.1.1 Density measurement

Density is calculated with the help of Archimedes' principle (eq.2.1and 2.2) the steps are described as follow

Step 1: Preparation of the Sample

- Ensure the casted 7075 Al sample is clean and dry.
- Remove any surface oxides or loose particles.

Step 2: Weighing in Air

- Weigh the dry sample in air using the electronic balance.
- Record the mass as Wair (in grams).

Step 3: Weighing in Water

- Tie the sample with a thin thread and suspend it fully in distilled water (without touching the beaker's sides or bottom).
- Ensure no air bubbles are sticking to the surface.
- Record the submerged weight as W water (in grams).

Archimedes' principle to calculate the volume:

$$V = \frac{W_{air} - W_{water}}{\rho_{water}} \quad (2.1)$$

Where ρ_{water} = 1 g/cm³ at room temperature,

After putting the measured value in equation 2.1

$$\text{Volume} = \frac{267-170}{1} = 97 \text{ cm}^3$$

$$\rho_{sample} = \frac{W_{air}}{V} \quad (2.2) \quad , \quad \rho_{sample} = \frac{267}{97} = 2.75\ gm/cc$$

2.1.2 Heat treatment

The sample was first subjected to solution heat treatment at 470°C for varying durations (2h, 4h, 6h, and 8h) in a muffle furnace (Fig. 2.4(b, c)), straight away followed by quenching at room temperature water (Fig. 2.4(d)). The optimal SHT condition was determined to be 470°C for a duration of 8 hours. Subsequently, aging was carried out for an entire period of 24 hours at two distinct temperatures (Fig.2.5(a, b)), 220°C and 140°C, at 3-hour intervals in the same furnace. For each of the two aging conditions, a total of eight samples 12 mm × 10 mm x 10 mm (Fig.2.4(a)) were produced. The aging process is optimized over 21 hours at 140°C.

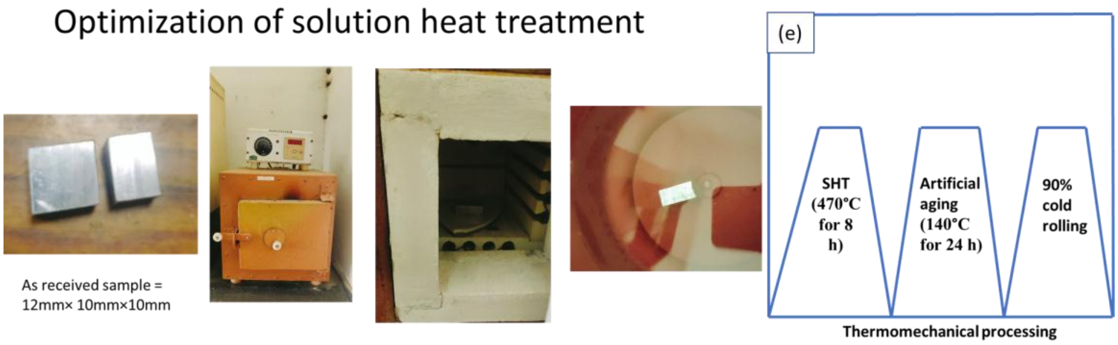

Fig. 2.4(a) As received sample (b, c) Muffle furnace, (d) quenched sample (e) Thermomechanical processing

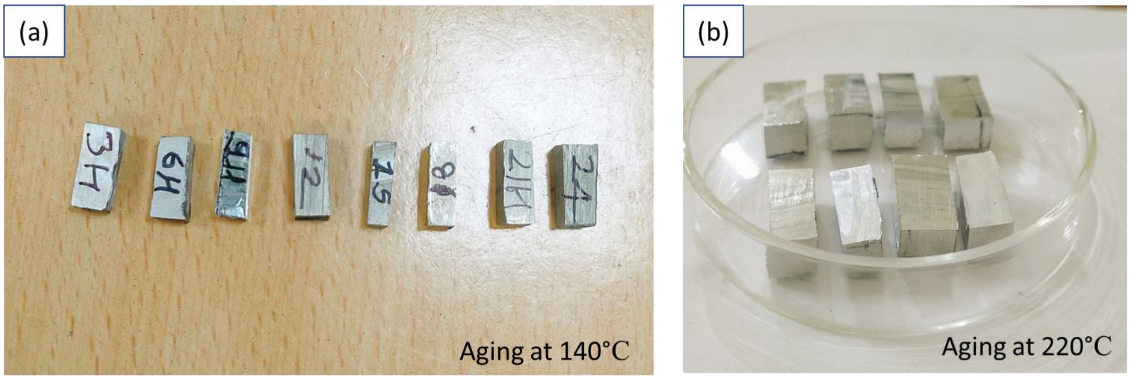

Fig.2.5 (a) Samples for aging at 140°C, (b) Sample for aging at 220°C

2.1.3 Thermomechanical processing

The rolling process had to be optimized next. A 75 mm roll diameter and 230 mm/min rolling speed were employed in the rolling machine (Fig.2.9 (f)). The sample was first cold rolled right after solution heat treatment (SHT), but after 45% distortion, it broke (Fig.2.7(a)). Furthermore, surface cracks at 60% deformation were produced by warm rolling (WR) the SHT sample at 200°C (SHT+WR) (Fig. 2.7(b)). At 30% distortion during the first cold rolling following SHT (SHT+CR), an alligator flaw was noticed (Fig.2.9(c)). In order to obtain a true strain of "$\varepsilon_t = 0.35$" at 30% deformation, the authors first made seven passes. Only 45% deformation was attained with SHT+CR (Fig.2.9(a)), even though the deformation was decreased per pass. However, the 7075 Al alloy was successfully cold rolled after peak ageing at 140°C for 21 hours, attaining a maximum deformation of 90% (SHT+PA+CR (90%))(Fig.2.8(b), (c), Fig.2.9(e)). A total of 16 passes were executed to accomplish 45% deformation, yielding a true strain of "$\varepsilon_t = 0.59$", and 17 passes were made to achieve 90% deformation, yielding a true strain of "$\varepsilon_t = 2.3$". Just 45% reduction was the highest, even with a gradual reduction in the first phase. 90% deformation was achieved through cold rolling in 7075 Al alloy thanks to the optimized procedure.

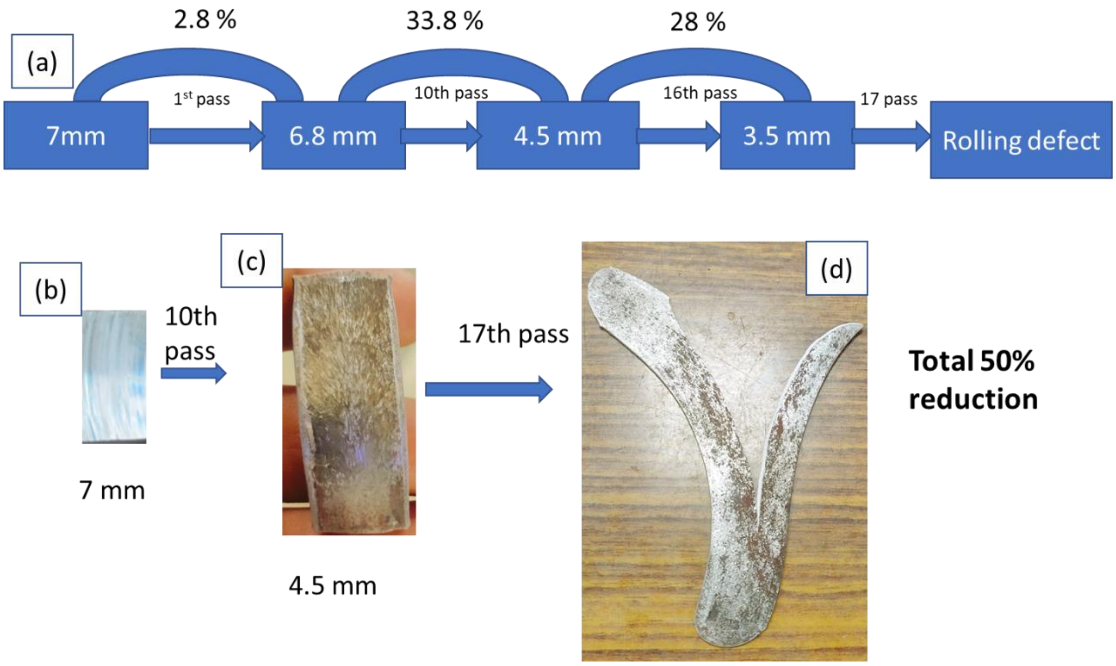

Fig. 2.6(a) Rolling process (b) as received sample (c) 35% deformed sample(d) deformed sample with rolling defect of center crack

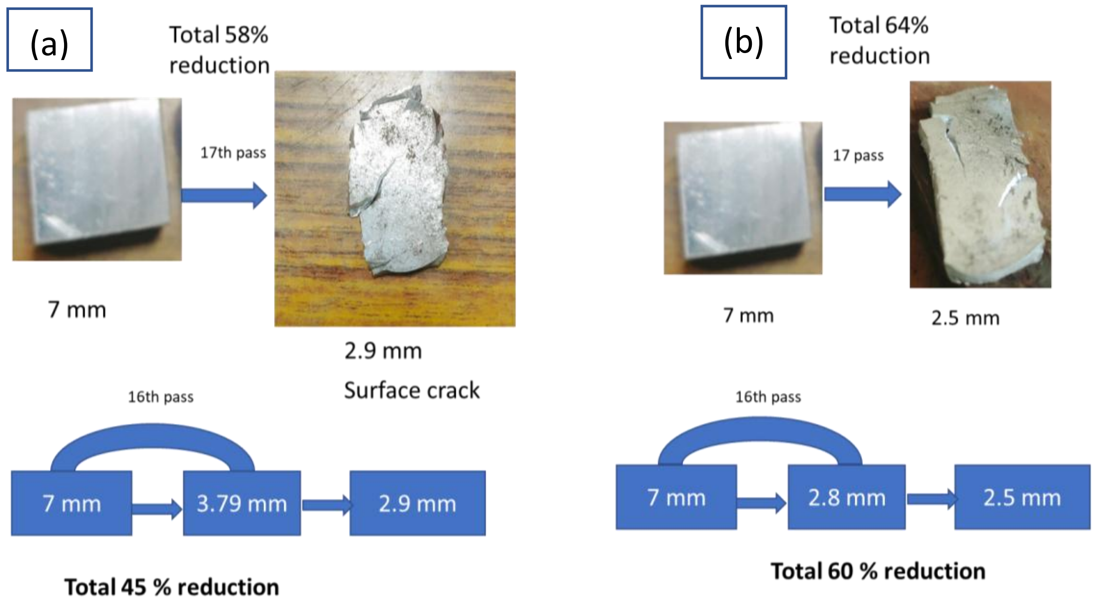

Fig. 2.7(a) cold rolling after SHT, (b) warm rolling after SHT at 200°C

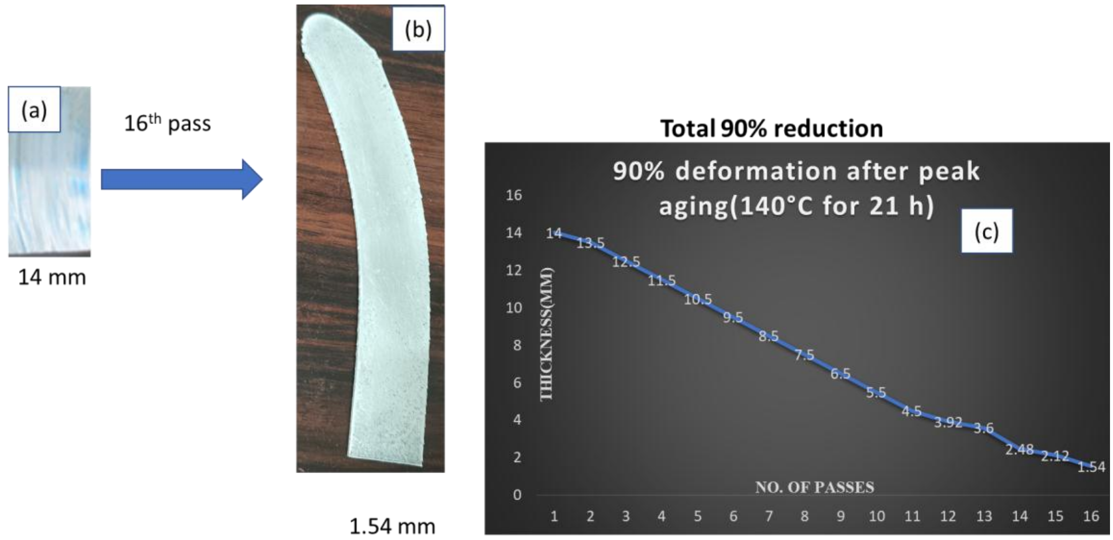

Fig.2.8 (a) PA sample, (b) 90% CR sample, (c) rolling graph for 90% CR sample

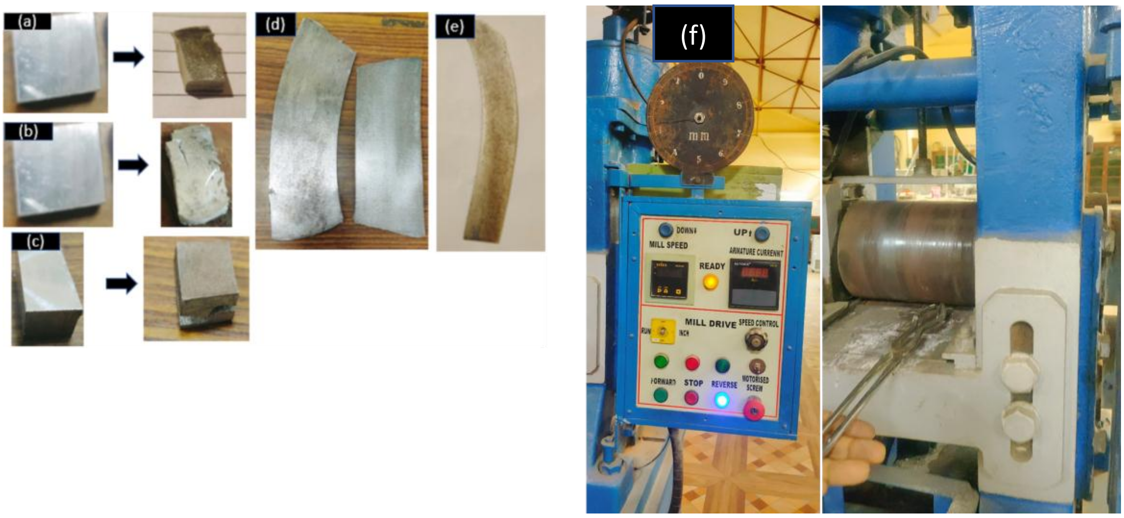

Fig. 2.9 (a) Cold rolling after SHT with 45 % deformation, (b) Warm rolling after SHT at 200◦C with 60 % deformation, (c) Alligator defect of SHT sample after 30 % deformation in cold rolling, (d) 80 % deformed sample after 21 h aging at 140◦C, (e) 90 % deformed sample after 21 h aging at 140◦C (f) Rolling machine.

2.2 Metallography investigation

2.2.1 XRD

After solution heat treatment to identify the precipitates' dissolution, XRD was executed. A sample with a cross-section of 10 × 10 mm² was created using a "highspeed diamond cutter" of Banbros company. The XRD was performed on a Rigaku company XRD machine (Fig.10) with a scan rate of one degree per minute, and the 2θ range was 20° to 90°.

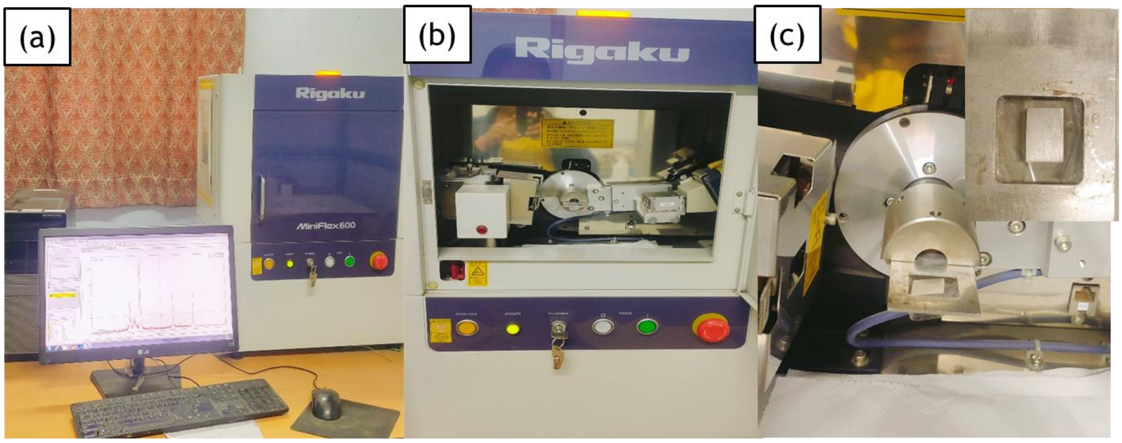

Fig.2.10 XRD machine, (b) vacuum chamber of XRD, (c) sample holder

2.2.2 Optical- microscopy

The "Banbros CMEX BOM-2000X" optical-microscope (Fig.2.11(b)) was employed to examine the 7075 Al alloy microstructure. Starting at P600 and working up to P800, P1000, P1500, and P2500, the samples were first polished using emery papers of various grit levels. The samples were then polished with cloth using (Fig.2.11(a)) diamond paste and diamond spray until they had a mirror-like sheen. Keller's reagent, which is composed of the mixture of "2.5% HNO_3, 1.0% HF, 1.5% HCl", and 95% H_2O, was used for etching.

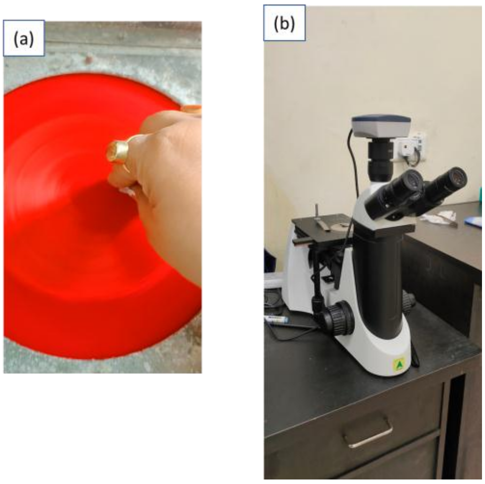

Fig. 2.11 (a) Fine polishing (cloth polishing), (b) Optical microscope

2.2.3 Scanning electron microscopy (SEM)

SEM was conducted on broken tensile and CT (compact tension) samples for a fractography research. The purpose of this fracture surface analysis was to examine the types and causes of fractures. This is also used to observe the surface of corroded samples. The corroded sample is mechanically polished with help of 2000 grit emery paper to remove the corrosion product. A SEM running at a “accelerating voltage of 15 kV” was employed in the experiment (Fig.2. 12).

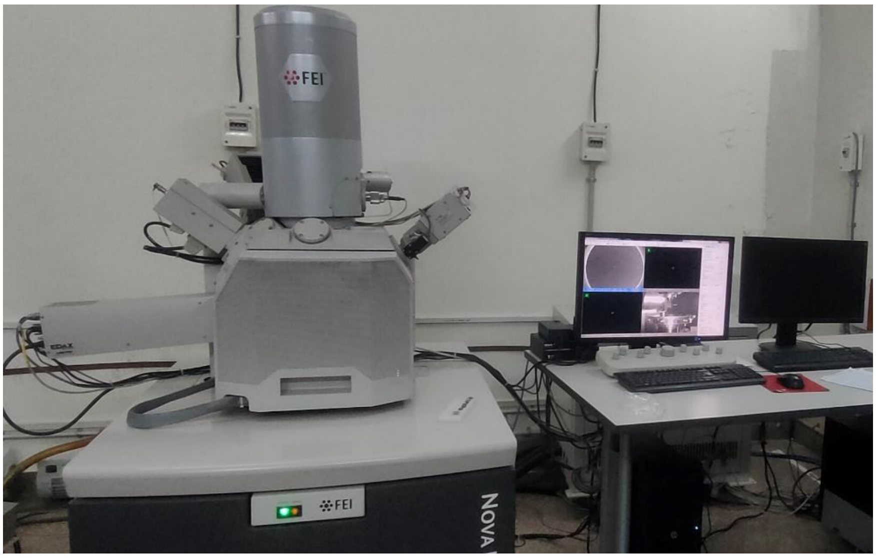

Fig.2.12 FE-SEM instrument

2.2.4 Electron Backscatter Diffraction (EBSD)

"The field emission (FE) scanning electron microscope Quanta 200", running at 25 kV with the TSL-EDX OIM system, was used to perform Electron Backscatter Diffraction (EBSD) in order to examine the microstructural characteristics. The distorted materials' texture and microstructure were examined. Mechanical polishing was used to bring the sample surfaces to a mirror-like finish for EBSD analysis in order to prepare them for EBSD measurement. The samples were prepared by polishing them with a variety of emery sheets, ranging in grit from P1000 to P2500, then polishing them with cloth, and then electropolishing them using a solution of 20% methanol and 80% perchloric acid to eliminate strain caused by these procedures. EBSD was then used to analyze this stress-relieved surface. A step size of 0.025 μm was used for samples that were distorted. The TSL-OIM program was used to do the EBSD analysis. The EBSD investigation's parameters in this work are in line with those documented in the literature [31,82,83]. Grain orientation spread (GOS) is a metric used to differentiate between deformed and dynamically recrystallized (DRXed) grains; deformed grains have large GOS values [31]. Since DRXed grains are not under stress, they must have low GOS levels. Even in magnesium alloy [30], recrystallized grains are frequently distinguished from deformed ones using a GOS threshold of 1° to 2° [32–33]. To distinguish recrystallized grains from other grains, we employed the following criteria in this study: "GOS < 1° and a boundary misorientation angle >15°". We used a criterion of GOS > 2° and a grain rotation angle between "2° and 15°" under all circumstances in order to separate sub-grains from IPF maps. While recrystallized grains have a border misorientation angle greater than "15°", sub-grains were expected to have a grain rotation angle between "2° and 15°"[84].

2.2.5 Transmission electron microscopy (TEM)

A peak-aged sample was subjected to TEM in order to examine the precipitate development process. The twin jet electro-polishing method was used to prepare the TEM sample. First, emery sheets with different grit sizes (1000 to 2500) were used to reduce the sample's thickness to 40 μm (Fig.2.13(a, b, c)). Disc grinder (2.13(f)) is used to reduce the sample thickness up to 30 μm. A 3 mm punching machine (Fig.2.13(e)) was then used to punch the sample (Fig.2.13 (d)). After that, polishing was done using a twin jet polisher (Fig.2.14) that had a sample holder for discs with a diameter of 3 mm. Double- nozzles, which functioned like cathodes, were placed between the disc of sample, which constituted the anode. The nozzles and the sample disc were both immersed in an appropriate electrolyte. Electrolyte streams were cycled through the nozzles on either side of the sample by a tiny submersible pump that was built into the apparatus. A blend of 20% perchloric acid and 80% methanol was utilized as the electrolyte. The temperature and voltage used for the polishing procedure were -20°C and 10V, respectively. Prior to the TEM analysis, the prepared sample was kept in a methanol solution. To guarantee an accurate assessment of hardness, the samples were polished to a mirror-like sheen for the hardness test.

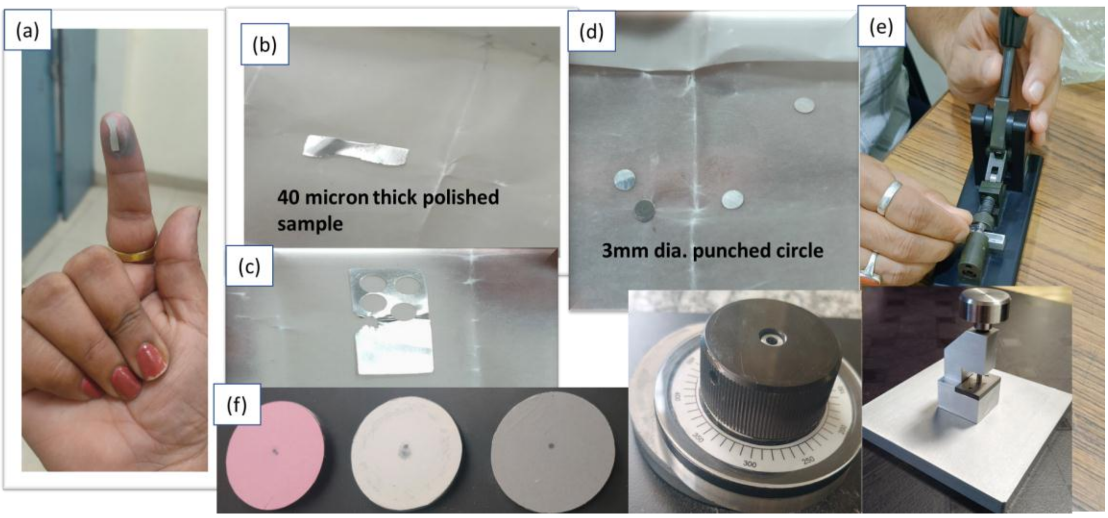

Fig. 2.13 (a, b, c) 40 μm thin sample, (d) 3 mm dia. punched hole, (e) Punching machine (f) disc grinder

Fig. 2.14 Twin Jet polishing setup

2.3 Corrosion testing

2.3.1 Immersion testing

The 7075 Al alloy samples were ultrasonically cleaned in ethanol and dried with cold air before immersion. They were then immersed in a 3.5% NaCl solution at 30 ± 1 ℃ for 120 hours in salt spray chamber (Fig. 2.15 (a, b, c, d)). To remove corrosion products, each sample was placed in concentrated nitric acid for 5 minutes, followed by washing with distilled water and drying in cold air. The five condition samples (SHT,

SHT+PA, SHT+60% WR, SHT+45% CR and SHT+PA+90% CR) in normal and 3.5% NaCl solution is shown in Fig. 2.15(e)

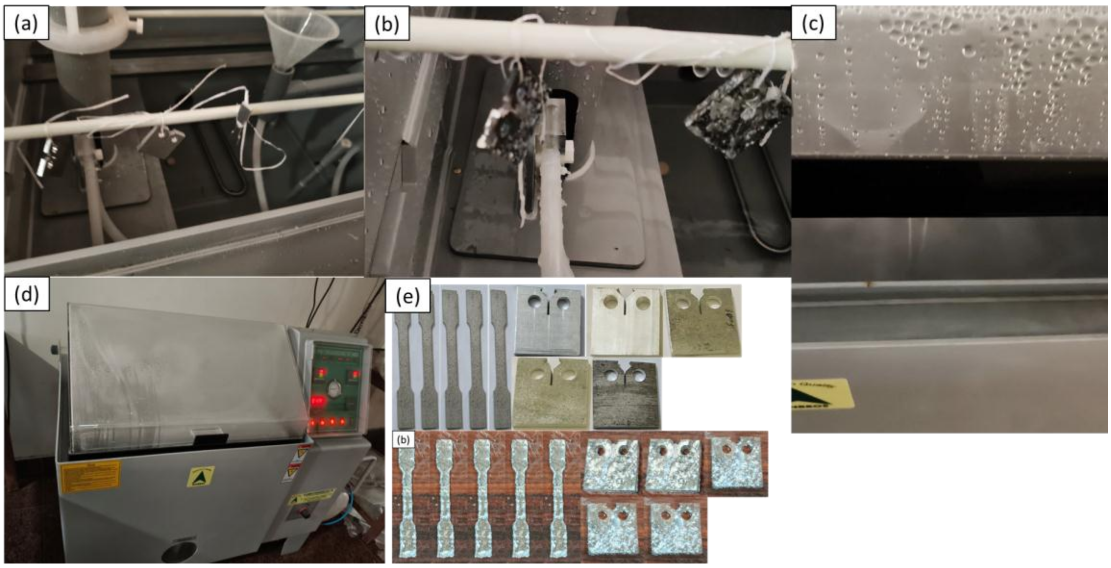

Fig. 2.15 (a, b) Hanged tensile and CT samples in salt spray chamber at normal condition and 3.5% NaCl solution condition, (c) NaCl fog, (d) salt spray chamber, (e) five condition samples (SHT, SHT+PA, SHT+60% WR, SHT+45% CR and SHT+PA+90% CR) in normal and 3.5% NaCl solution

2.3.2 Electrochemical testing

Electrochemical testing was conducted using an electrochemical workstation (Gamry Interface 1010 E Fig. 2.16(a)) connected to a three-electrode cell (Fig.2.16(b)). The tests were carried out in a 3.5% NaCl solution at 30 ℃. A rectangle sample with an exposed area of 1.0 cm^2 served as the working electrode, while a thin platinum electrode and a saturated silver electrode (Ag /AgCl) functioned as the counter and reference electrodes, respectively. Once the open circuit potential stabilized, the potentiodynamic polarization curve was recorded at a scanning rate of 0.5 mV/s over a potential range of −0.8 V to 1.5 V relative to the open circuit potential. The experimental data were analyzed using Gamry Echem Analyst. Electrochemical impedance spectroscopy (EIS) was performed at the open circuit potential with a sinusoidal perturbation signal of 10 mV, spanning a frequency range from 100 kHz to 0.01 Hz. Gamry Echem Analyst software was used for data analysis. Corroded five conditions sample in 3.5% NaCl solution are illustrated in Fig. 2.16(c).

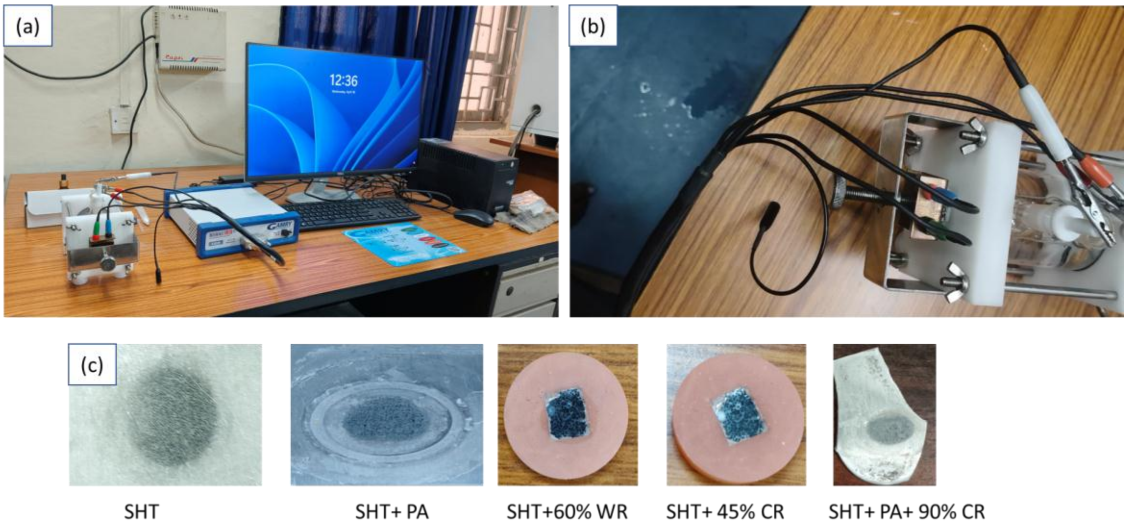

Fig.2.16 (a) Gamry potentiostat and EIS set up, (b) electrochemical cell (c) Corroded five conditions sample in 3.5% NaCl solution

2.4 Mechanical Testing

2.4.1 Vickers Hardness test

For hardness test, samples were mirror like polished (Fig.2.17(a)) for the proper hardness investigation. The Vicker's hardness tester (Fig. 2.17(b), (c)) was employed to assess the hardness of samples. During the hardness measurements, specific parameters were utilized, including a 10 kgf load and a 10-second dwell time. To ensure accurate results and prevent the influence of local plastic deformation caused by indentations, a minimum distance was maintained between the two Vicker's hardness indentation spots. The average hardness value was determined by taking the average of 7 measurements. The hardness samples were extracted from the central regions of the processed samples. This test involves applying a controlled force, typically using a pyramid-shaped diamond indenter with a square base (Fig. 2.17(b), (c)), to the surface of the material being examined. The force was usually applied for a specific duration, and then the resulting impression or indentation was measured.

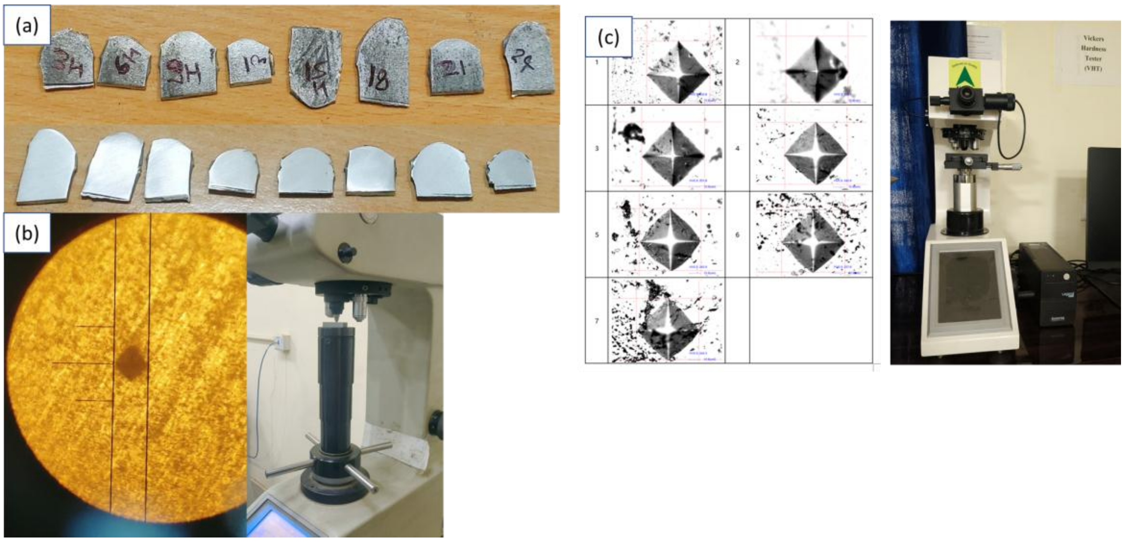

Fig.2.17 (a) Before and after polished sample, (b) Vickers hardness tester with square indent, (c) micro-Vickers hardness tester with 7 square indentations.

2.4.2 Tensile test

Tensile test specimen was prepared according to ASTM E8M [85–87] (Fig. 2.18 (a)) with the help of EDM wire machine. 3 specimens of each condition (SHT, peak aged and 90% cold rolled after peak aged) were prepared to take average of the results of all three specimens in all three conditions. Tensile test was performed on Instron tensile testing machine 25 KN capacity shown in Fig. 2.19(a). Strain rate used during the tensile test was 1×10^{-3}/sec in each condition.

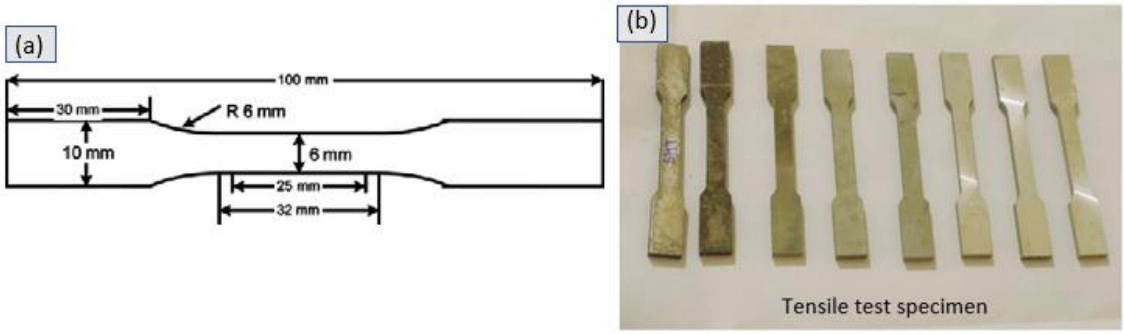

Fig. 2.18 (a) ASTM E8M tensile test specimen drawing, (b) finally prepared tensile test specimen for three conditions (SHT, peak aged and 90% cold rolled after peak aged)

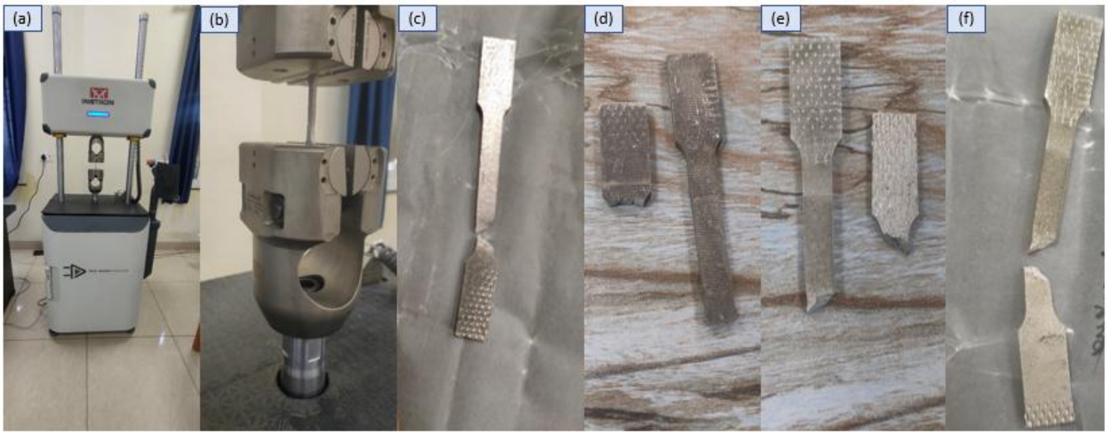

Fig.2.19 (a) Tensile testing machine, (b) dog bone specimen in tensile grip, (c) As received, (d) SHT, (e) Peak aged (SHT+PA) (f) Rolled (SHT+PA+90% CR) fractured samples

2.4.3 Fracture test

Compact tension (CT) test was prepared according to ASTM E647 [43,56,57,88] (Fig. 2.20(a)) with the help of EDM wire. Three specimens were prepared for each condition (SHT, peak aged, and 90% cold rolled after peak aged) in order to calculate the average results across all three specimens for each of the three conditions. Fracture test was executed on the same machine on which tensile test was performed (fig2.21(a)) with different grip displayed in Fig. 2.19 (a). In a typical J_{1c} fracture test, the test specimen was loaded in a controlled manner while a crack was initiated and then allowed to propagate through the material. All fractured samples of tensile test and fracture test were preserved carefully for study of fractography.

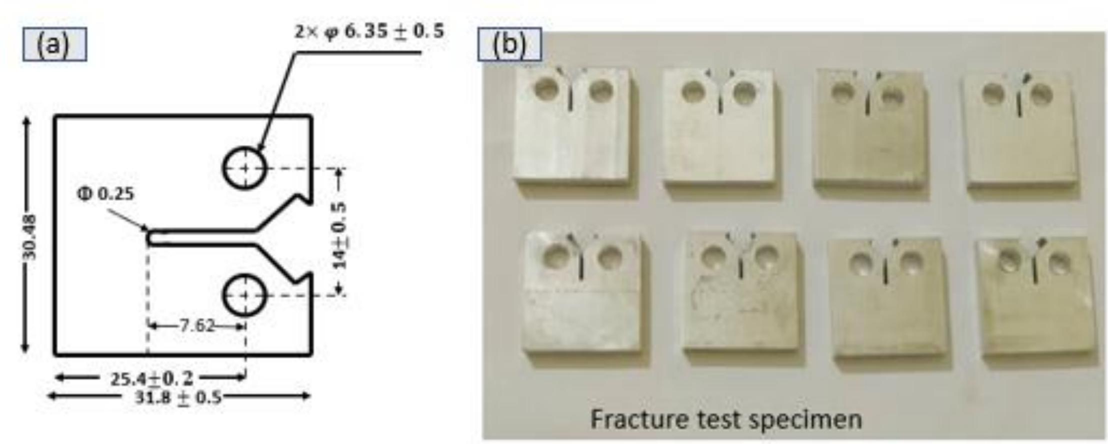

Fig. 2.20 (a) ASTM E647 CT (compact tension) test drawing (all dimensions are in mm), (b) fracture test specimen.

Fig. 2.21 (a) Grip for fracture test, (b) and (c) are the fractured CT sample by J1c process.