EFFECT OF AGEING ON MECHANICAL AND FATIGUE BEHAVIOUR OF 2014 ALUMINIUM ALLOY

A DISSERTATION

Submitted in partial fulfillment of the

Requirements for the award of the degree

of

MASTER OF TECHNOLOGY

in

MECHANICAL ENGINEERING

(With Specialization in Machine Design Engineering)

by

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CANDIDATE'S DECLARATION

I hereby declare that the work carried out in this report entitled **"EFFECT OF AGEING ON MECHANICAL AND FATIGUE BEHAVIOUR OF 2014 ALUMINIUM ALLOY"**, is presented on behalf of partial fulfillment of the requirement for the Dissertation of "**Master of Technology**" in Mechanical Engineering with specialization in **Machine Design Engineering** submitted to Department of Mechanical and Industrial Engineering, Indian Institute of Technology Roorkee under the supervision of **Prof. I. V. Singh,** professor and **Prof. B. K. Mishra,** Professors, Department of Mechanical and Industrial Engineering, Indian Institute of Technology Roorkee.

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This is to certify that the above statement made by the candidate is correct to the best of my knowledge and belief.

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ABSTRACT

This work deliberates the influence of ageing on Mechanical and Fatigue properties of Aluminium alloy 2014T6. In addition, the influence of ageing on micro-structure is also considered using Optical Microscopy, Scanning Electron Microscope (SEM) and X-ray Diffraction. Experiment begins with solution treatment to the as received Aluminium alloy 2014 T6 (industrial available) at temperature 504°C. Solution treatment restore any microstructure alterations present. Quenching followed by solution treatment locks the microstructure of AA2014 in Super saturated meta-stable state. Subsequently, ageing treatment was completed at three different temperatures and four different time periods. No significant variations detected in mechanical and fatigue properties for ageing temperature of 130°C. however a decent hike was observed for ageing at temperature 160°C. The raise was continuous up to 10Hour of time period. Ultimate Tensile Strength and Yield Strength for this ageing parameter was 490MPa and 440MPa with an increase of about 35% and 50% respectively. An increase in fatigue behaviour was also observed till 8Hour of ageing after that a deleterious effect was noted. However, ageing has a deleterious effect on material elongation properties. Exploring microstructure with different tricks shows nucleation and growth of different phases while ageing of alloy. SEM, EDX and XRD plots of differently aged samples discloses that as the number and size of the phases increases the material strength increases too. Examining the crack through Optical and SEM reveals the deviation of crack path from straight line to zig-zag along with ageing. Investigations concerning crack path reveals that zig-zag path are the mark of increase in fatigue resistance. The deviations are due to the presence of hard phases nucleated during ageing. Ageing at 190°C shows increase in material strength for a while later on it has a deleterious effect on mechanical properties. This may be a result of over ageing of the alloy. The number and sizes of the phases present in these cases are relatively larger than 160°C ageing.

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Chapter 1

INTRODUCTION AND LITERATURE REVIEW

1.1 INTRODUCTION

The age hardening Aluminium alloys have been always the back bone of the aircraft industries. Its strength to weight ratio has been always the prime driver for its selection as the aircraft material. since the first days of its introduction, Aluminium always have been the subject of numerous research studies about its reduction of weight to strength ratio. Aluminium have not only the benefit of low weight to strength ratio but it also comes with some important properties like Corrosion resistances, high electrical and thermal conductivity, which allow to it use in a broader use like automobile parts, general home appliances, electronics circuits etc. Earlier of development of Aluminium alloys comes with hit and trial method but after a several years of research of microstructure and properties engineers have developed several successful methods to enhance the properties of Aluminium alloys like Cryo-Rolling, Equal Channel Angular Processing (ECAP), High-Pressure Torsion (HPT), Repetitive Corrugation Method, precipitation methods etc. A number of researches had been carried out to know about the aluminium alloys, their properties, microstructure, fatigue life, corrosion resistance, crack growth, machinability, weldability and others. A literature review carries important notes of previous researches and give a foresight to new researchers.

Here are some of the researches published related to the aluminium alloys properties improvements. (especially aluminium alloy 2014).

1.2 LITERATURE REVIEW

E. A. Starke *et al.* (1996) They studied the structure, various load on the structure and their properties requirement by the structure of different Aluminum aircraft alloys and describes the various mechanical ways for the development of modern Aluminium alloys to improve performance. Apart from these his work also includes some of the mechanical treatment like precipitation, stretching method, rolling method to improve the mechanical properties of 2XXX, 7XXX and 8XXX aluminium series.

T Gladman *et al.* (1998) They described the interaction of dislocation with the hard and soft dispersion particles. The hard-dispersed particles do not allow the dislocation to cut it and thus they by-pass it by Orowan bow mechanism. While interacting with the hard-dispersed particles the dislocations end up with a close loop around the particle. The dispersion particles are hard at a longer time or high temperature of ageing. The softer particles are sheared by the dislocation by various mechanisms like Coherency Effect, Modulus Effect, Interfacial Effect, Ordering or Anti-phase or Staking Fault energy. The energy or factor associated with the different sheared mechanisms <u>are</u> responsible for the increase in the strength.

Rafiq A. Siddiqui *et al.* (1999) They studied the effect of ageing treatment on AA6063 through tension and fatigue testing. The ageing was done for 373, 398, 423, 448, 473 and 498K for a time period of 2, 4, 6, 8,10 and 12Hour. Experimental details revealed that 8 to 10Hour at temperature 448K was the most suitable combination for the ageing of the aluminium alloy 6063. The precipitation size increases with increasing time or temperature. the Mechanism of precipitate development was ‘solid solution ➡ Guinier-Preston zone ➡ precipitate formation’. The fatigue resistances increase with temperature and time and yield at 473K for 6Hour.

Y Totik *et al.* (2004) Studied the effect of solution treatment temperature on hardness and torsion strength for AA2014 and AA6063. For a solution treatment temperature range of 150℃ to 500℃ the results show that the hardness value decreases with increases temperature. The optimum condition for solution treatment based on the torsion result was found to be 470℃ for 8Hour.

R Sadeler *et al.* (2004) Studied the effect of different temperature solution treatment followed by ageing on the mechanical behaviour through rotating bending fatigue test. Solution treatment were done at 410, 450, 480 and 510℃ followed by quenching in water. The solution treated sample further aged at 190℃ for 7Hour. About 43%

improvement was observed in fatigue result in comparison to cast alloy upon as a result of solution treatment at 510℃ followed by ageing.

T. J. Bastow *et al.* (2006) They investigated that the GP zone formation proceed rapidly even at room temperature. Their work reveals that the diffusion of Cu atoms from solid solution complete within 50Hour. Only a small residual amount of Copper remains in the solid solution as a meta-stable state.

Hulya Kacar *et al.* (2007) they investigated the age hardening behaviour of self constructed Aluminium alloy 2014 through powder metallurgy. The variation in hardness was observed by varying the sintering temperature and the aging temperature. 60% increases in hardness were observed on ageing at 473K comparative to natural aging.

C V Singh *et al.* (2010) they studied the formation of precipitation in aluminium alloys and also simulate them. They found that the precipitation formation begins with the nano-size particle formation named as Guinier-Preston particles. These particles are coherent in nature initially. The GP zones particles act as restriction in the dislocation movement and there for the strength of the material increases. However, after a time period the precipitate become incoherent with material and the strength of material start decreasing.

Sushanta Kumar Panigrahi *et al.* (2011) Investigated the effect of ageing on room temperature and cryo-rolled Aluminium. The investigation includes microstructure study and mechanical properties of aluminium alloy 7075. They observed improvement in properties after ageing of cryo-rolled sample. The optimum ageing time found to be 100°C for 45 Hours.

Supriya Nandy *et al.* (2015) Investigated the influence of ageing on 6063 aluminium alloy. The temperature of ageing was 175℃ for a period of 10 minute to 14 days. The result obtained shows that till 8Hr the hardness value increases and then it starts decreasing. The peak condition was identified as 175℃ for 8 Hour. Besides the mechanical behaviour they also investigated fracto-graph.

Yashpal Kaushik *et al.* (2015) studied the differences properties of aerospace material including creep effect, fatigue and fracture. The result shows that AA are best suited for aircraft seats. Among AA 8XXX series are best suited due to its low density.

They found that the creep behaviour of the plate is slightly better than the sheets. The creep behaviour is very sensitive to the chemical composition of the Aluminium Alloys. Their experiments reveal that the AA2024 have excellent fatigue and fracture properties. There is no effect of higher ΔK on crack propagation whereas low ΔK decreases the fatigue crack propagation rate.

Suresh Chandra *et al.* (2016) Investigated the hardness property of AA2014 processed by aging at 183°C for 2Hr, 4Hr, 8Hr, 10Hr and 12Hr. He also studied the microstructure of the aged material through optical and XRD. The peak condition found at 183°C for 8Hr. The hardness value obtained at this temperature 129VH. He indicated the presence of the $CuAl_2$ phase during aging. The $CuAl_2$ represented as θ''. The precipitation hardening mechanism was followed by a sequence SSSS. *ie* α (Al) then α+θ'' followed by α+θ' then α+θ. Their experiment shows that as the number of participate increases the hardness value increases up to peak condition and then it start decreasing.

Amit Joshi *et al.* (2016) investigated the precipitation sequence through soft annealing after cryo-rolling on aluminium alloy 2014. Cryo-rolling reduction up to a true strain of 2.3 has been studied followed by annealing in temperature range of 373K to 723K for a time period of 45 minute. Peak strength was observed at CR+SA at 75% reduction and 373K.

P Rambabu *et al* (2017) they investigated the fatigue strength of different aluminium alloy. Their investigation reveals the optimum to AA2024 and 7075. They also reveals that the FCG behaviour of AA2XXX series are superior than 7XXX series. Apart from this they also studied different component and the material used in it.

Amit joshi *et al.* (2017) studied the behaviour of Aluminium alloy 2014 affected by cryo-rolling followed by annealing through high cycle fatigue test. Highest strength 122MPawas observed at CR+SA at 373K in high cycle fatigue.

1.3 Scope of work

Clearly, based on the literature review, Aluminium always have been the most demanding material in aerospace and automobile sector due to its light weight and high strength properties. AA with 4-4.5% copper have been always the primary material for the aerospace sector. Several attempts have been taken to improve the mechanical properties of the AA, but still there is a question of its optimum properties. There is a lot of mechanical and thermal processes to improve the mechanical properties on which work can be done.

- ✧ Effect of annealing and followed by aging treatment can be studied on the fatigue crack growth behaviour of aluminium alloy.
- ✧ A comparison of RT, CR, WR on the mechanical and fracture behaviour can be done and evaluated.
- ✧ Fracture toughness can be optimized for different warm rolling temperature.
- ✧ FCG behaviour can be simulated for the alloys.
- ✧ Creep and Corrosion behaviour of the Alloys under different temperature can be investigated.
- ✧ Forging treatment can be performed for the alloys at elevated temperature.
- ✧ High and low cycle fatigue and fracture properties can be evaluated at high temperature.
- ✧ Dynamic fracture toughness of Aluminium alloy can be investigated.
- ✧ Heat flow simulation of aluminium alloy can be done for annealing.

Chapter 2
Experimental details

2.1 MATERIAL SELECTION

The world-wide increased demand of the energy has convinced the aerospace industries to consider different ways to reduce the energy consumption. The selection of different material to reduce weight, dependency on the renewable sources of the energy, re-designing the engines for low fuel consumption are few of the affords. For the aerospace industries the strength to weight ratio was the primary driver for selection of the material. The commencing era of aerospace industries have not very much metal in use due to its weight to volume ratio. They have been using the woods. the use of aluminium are first used by Wright Brothers. However, there is not much role of the aluminium until the precipitation hardening and some of the others mechanical hardening technique are discovered. The age hardening technique was discovered by Alfred Wilm.

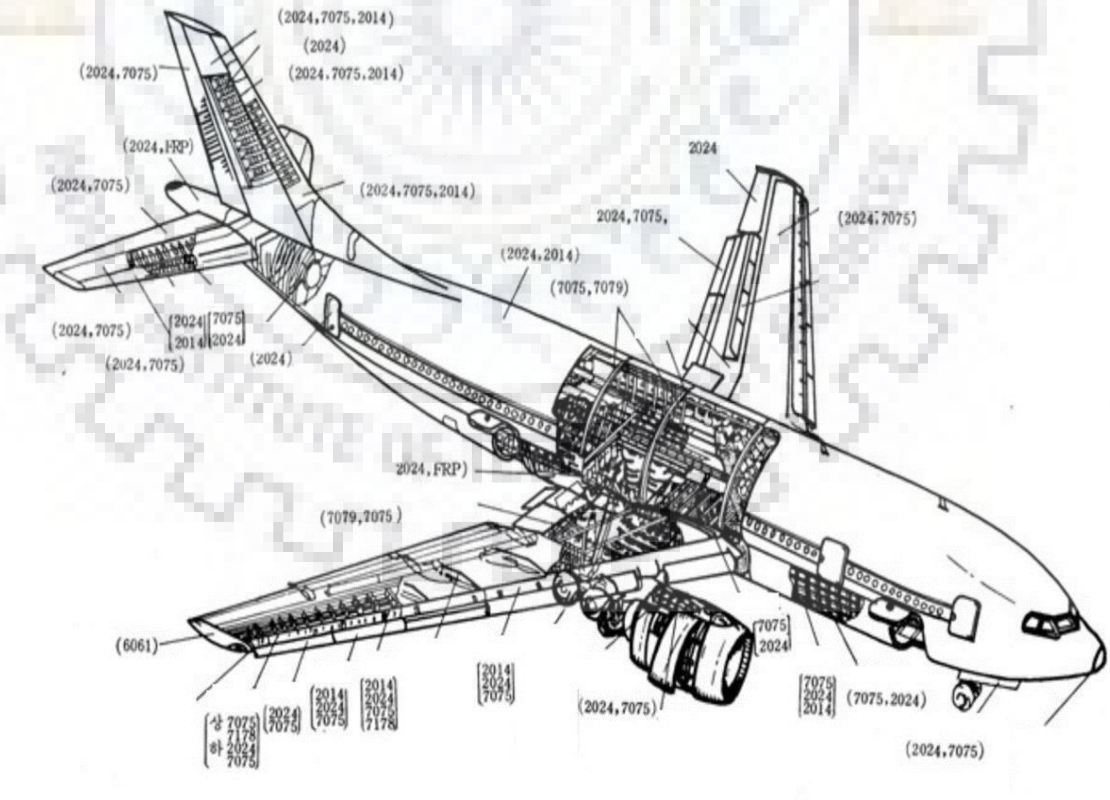


Fig 2.1 Various aluminium alloys with their used in an aircraft [3]

However, few recent material properties improvement technique like Cryo-Rolling, Equal Channel Angular Processing (ECAP), High Pressure Torsion (HPT), Repetitive Corrugation Method have drastically increased the use of the aluminium alloys in the aerospace industry. Around 40% of the material used in the aerospace are aluminium alloys.

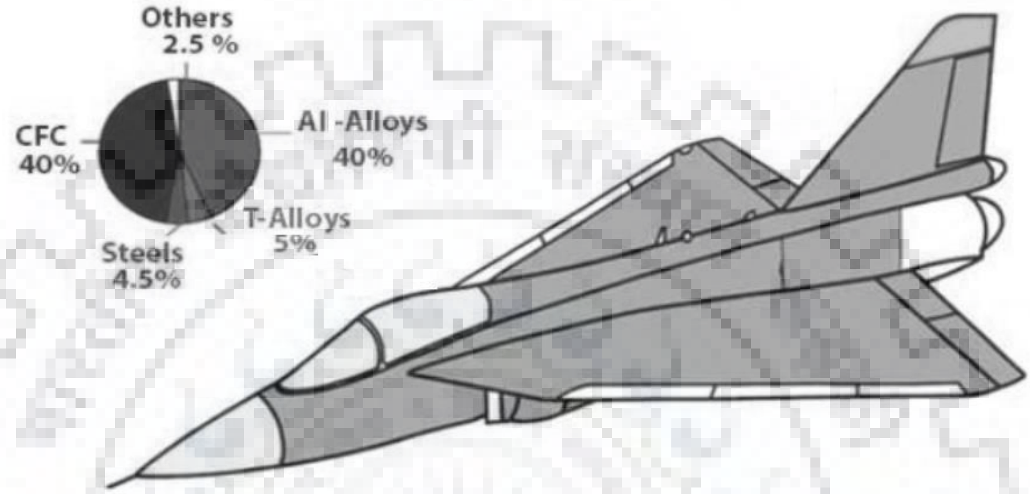


Fig 2.2 Estimated percentage of the material used in different defense industries or aerospace industries [4]

Figure 2.1 shows the different grades of aluminium alloys used in the aerospace industries. 2XXX aluminium alloys are the primary aluminium alloys for the aerospace industries. A recently development 7XXX aluminium series are also in demand due to its strength. 2XXX aluminium alloys are the strongest aluminium alloys in comparison to other aluminium alloys. 2XXX series consist of mainly copper as alloying element in aluminium. In this work aluminium alloy 2014 is selected for the different mechanical analysis based on the availability. AA2014 was obtained from Gayatri Industries India. Chemical composition of the obtained material are as follows

Table 2.1 Chemical composition of AA2014T6.

Element	Cu	Mg	Si	Mn	Fe	Ti	Zn	Ni	Aluminium
Composition	4.4	0.43	0.67	0.7	0.18	0.05	0.06	0.05	93.46

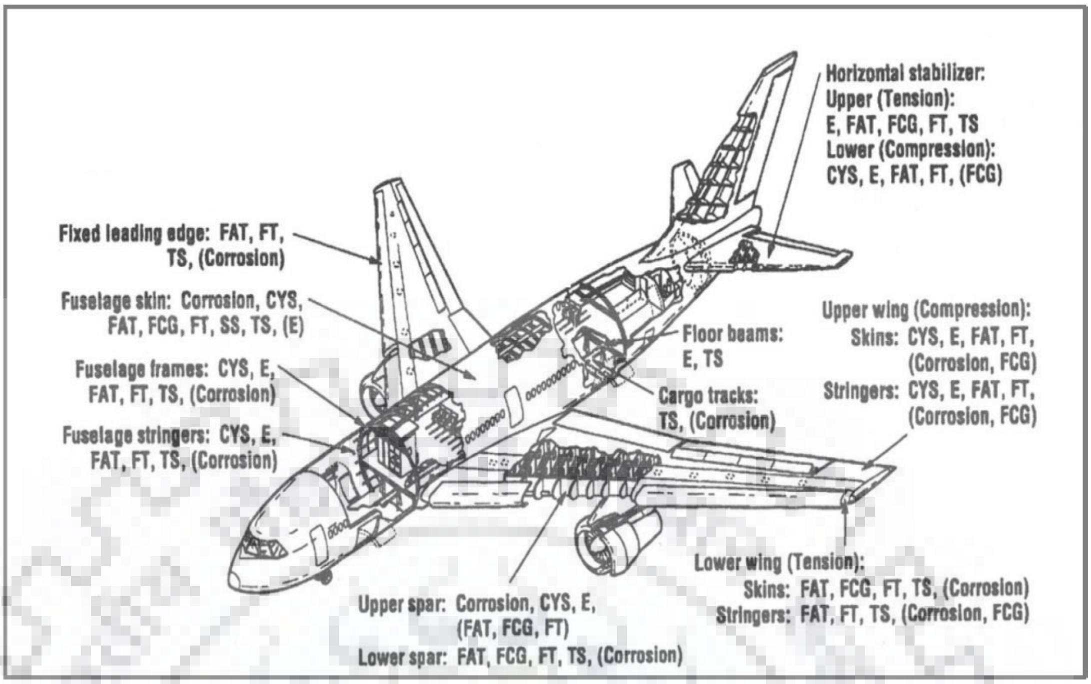


Fig 2.3 Structure of aircraft having different types of loads and the properties requirement of the different structures; FAT- fatigue; FCG- fatigue crack growth; FT- fracture toughness; TS- tensile strength; CYS- compressive yield strength [5] .

2.2 SOLUTION TREATMENT

The as received AA possess non-uniformity in nature in context of microstructure and its mechanical properties. The result obtained from these sample are quite inaccurate and are not stable. Solution treatment is a process of reducing segregation. It homogenizes the material through-out. This process consists of holding the material at a constant temperature then quenching it in room temperature water. Every material has its own solution treatment temperature. Holding the material at the temperature remove any dislocation density present in the material. Quenching in water lock the material state and make it stable. The quenching results SSSS (super saturated solid solution). A solution treated material contains single phase α through-out. Solution treatment provide equiaxed grains which makes the material easily machinable.

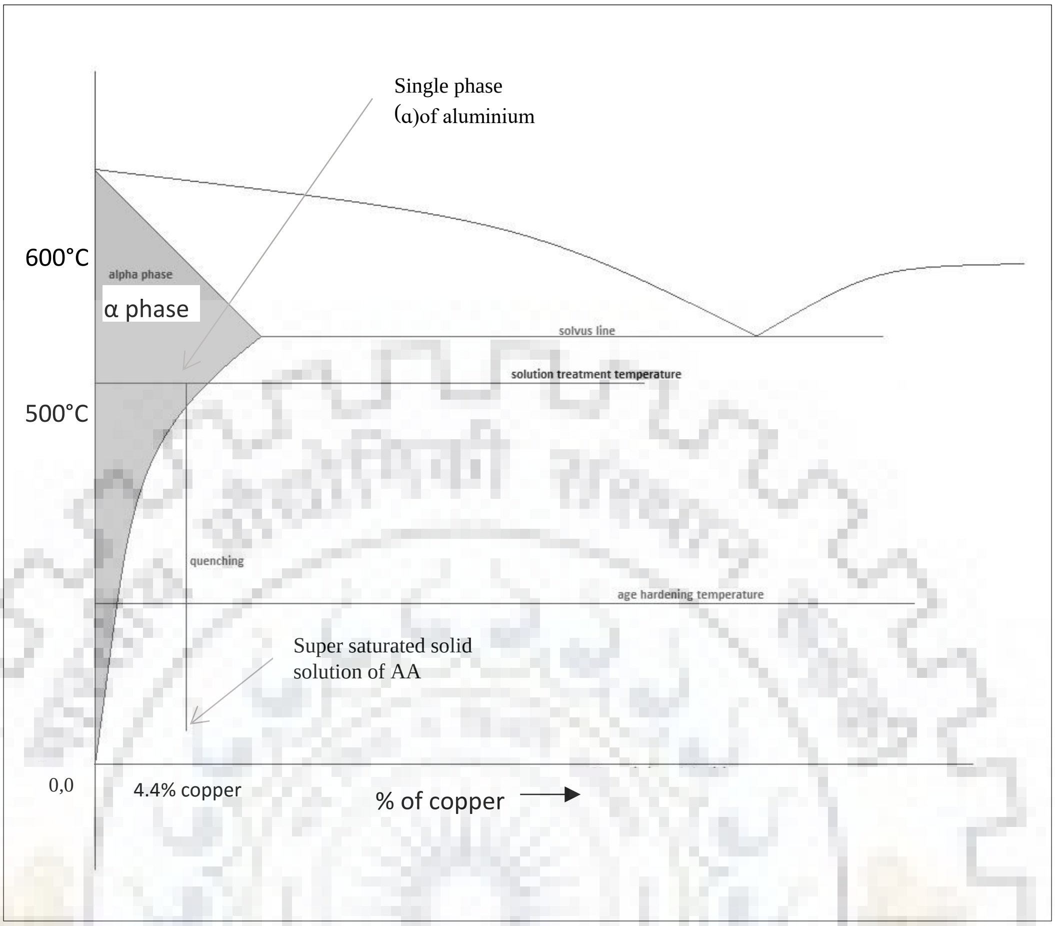


Fig 2.4 Phase diagram of Aluminium-Copper regarding solution treatment temperature.

For the Aluminium alloy the temperature is selected as 504℃ based on literature review and the Aluminium copper phase diagram. The holding time at this temperature is about 120 minutes. It was quenched in the running water. The solution treatment results the material into metastable single phase of copper locked in aluminium crystal.

2.3 AGEING

Ageing is a phase change phenomenon over time. There are various metal and materials whose properties changes over time period. The analysis of the material properties, phases, different structures over the time period comes under ageing treatment. Ageing of material over environmental condition for a long time period are termed as natural ageing. The ageing treatment done with high temperature and shorter time period are artificial ageing. Artificial ageing completed in three steps,

1. Solution treatment
2. Quenching

3. Age hardening

At first the material is heated to a temperature just below its solvus temperature and hold it till all the material comes in a single phase. Since all the material structure are in single structure therefor it is named as alpha structure. This procedure is called as solution treatment. The second step is to quench the material to locked the material in solid solution state. At high temperature the solubility of the material is very high. Upon quenching the material, the saturation solubility decreases and hence the material state become super saturated solid solution. The third step is to re-heat the material at the ageing temperature, hold it for the time and then air cool it. The metastable copper at high temperature form precipitate and increase the material strength. However, the precipitate growth keeps on increasing with temperature, the spacing between them keep on decreasing, the precipitates diameter keep on increasing. After a point the strength of the material start decreasing.

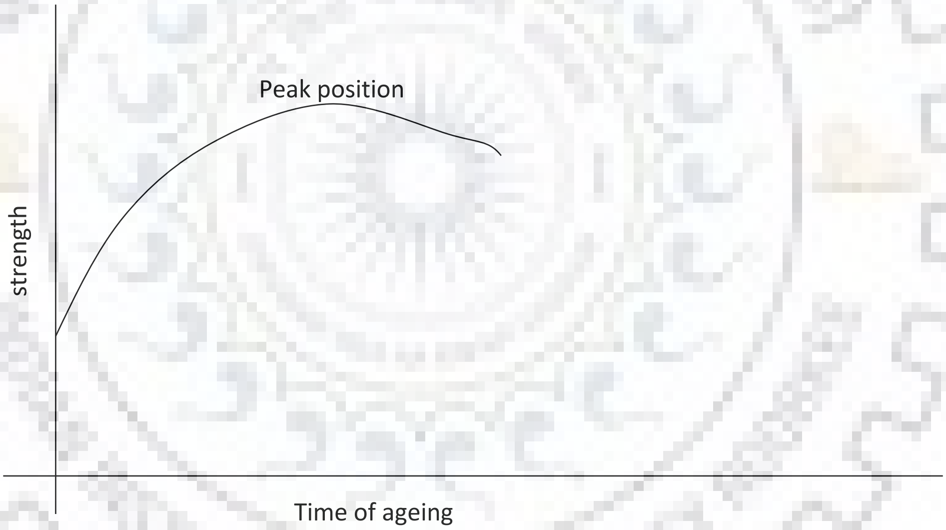


Fig 2.5 A normal ageing effect on the aluminium alloys. Strength found to increase initially and then it decreases.

Theory behind strengthen mechanism of precipitation hardening. There are two types of theories behind the precipitation hardening mechanism. The microstructural changes which take place during age hardening proceed with nucleation and growth of Coherent GP zones.

The zones are the precursors of particles which evolve through various intermediate stages, each governed by its own metastable phase diagram, towards a final equilibrium precipitate. The shearing effect takes place in this regime.

The degree of coherency falls as the particles coarsen, until the widely-spaced, incoherent particles of the overaged state are reached. The by-passing mechanism takes place in this regime.

2.2.1 Cutting mechanism/shearing mechanism the dislocations are assumed to cut/shear the dispersed particles. There are various explanations related to this theory as follows.

1. **Size/ coherency effect** this theory is defined over the lattice size of the precipitate to the lattice size of the matrix. The difference in the lattice parameter is counted for the increase in the strength of the material. The coherency factor is given by as

$$\varepsilon_{coh} = \frac{\alpha_p - \alpha_m}{\alpha_m}$$

Where α_p is the lattice parameter of the precipitate particle and α_m is the lattice parameter of the matrix around them.

The change in strength due to the precipitation hardening is given by as

$$\delta = 7 \times \varepsilon_{coh}^{1.5} \times G \times \left(\frac{r \times f}{b}\right)^{0.5}$$

Here r/b is a term representing the size of the precipitate with respect to atom size of the matrix and *f* representing the volume fraction of the precipitate.

2. **Modulus effect** the change in the modulus is responsible for the change in the strength of the material. The equation is as follows for the strength change.

$$\varepsilon_{mod} = \frac{G_p - G_m}{G_m}$$

Where G is showing the modulus effect of the corresponding precipitate and matrix phase. The change in the strength is given by

$$\delta = 0.01 \times \varepsilon_{mod}^{1.5} \times G \times \left(\frac{r \times f}{b}\right)^{0.5}$$

3. **Interfacial effect/chemical effect** This theory counts the extra surface generated by the dislocation shear movement. The extra surface generated need the interfacial energy. This interfacial energy of the precipitate particles is very high and the consumption of this energy strengthen the material.

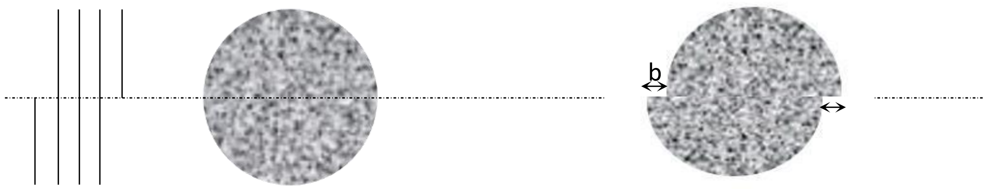


Fig 2.6 Dispersion phase particle interaction with the dislocation movement and the new area generated by them.

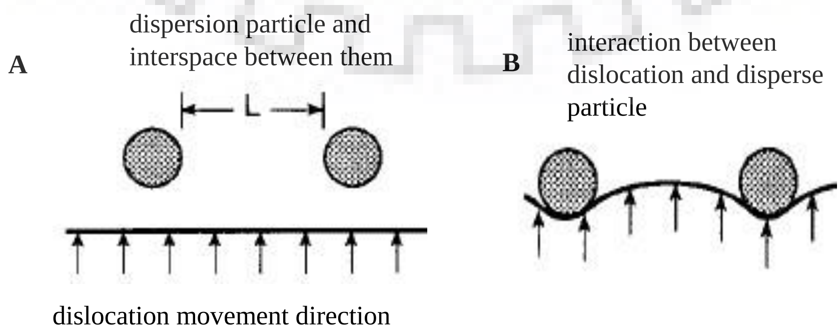
figure 2.5 shows how the extra area generated during the dislocation movement in the dispersion phase particles. The extra region and the interfacial energy related to them have a contribution to the strengthen mechanism as follows.

$$C_h = \frac{\gamma_s}{G \times r}$$

where γ_s is the surface energy, G is modulus of matrix and r is the size of the precipitate. The change in the strength is given by following equation

$$\delta = 2 \times \varepsilon_{ch}^{1.5} \times G \times \left(\frac{r \times f}{b}\right)^{0.5}$$

2.2.2 Bow mechanism/by-pass the disperse particles when the precipitate particles become large the dislocation particle are no longer to cut through them. They start bypassing them by various ways. These mechanisms include bowing, climb and dislocation slip mechanism. This process of by-passing of dislocation is called as Orowan Bowing mechanism.



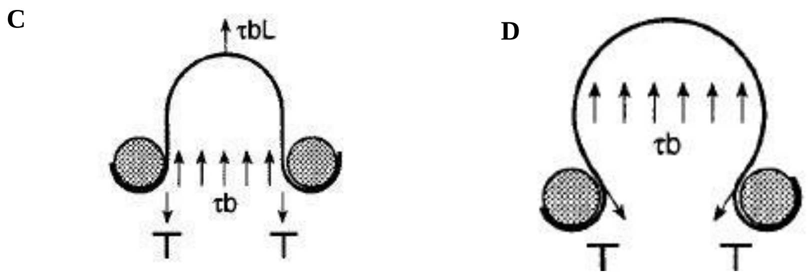


Fig 2.7 Dispersion hard particle interaction with the dislocation movement; (A) dispersion particle in the movement of dislocation; (B) the hard particles are unable to shear; (C) a by-pass formation of dislocation motion; (D) the dislocation by-passes and a close loop around the particle. [5]

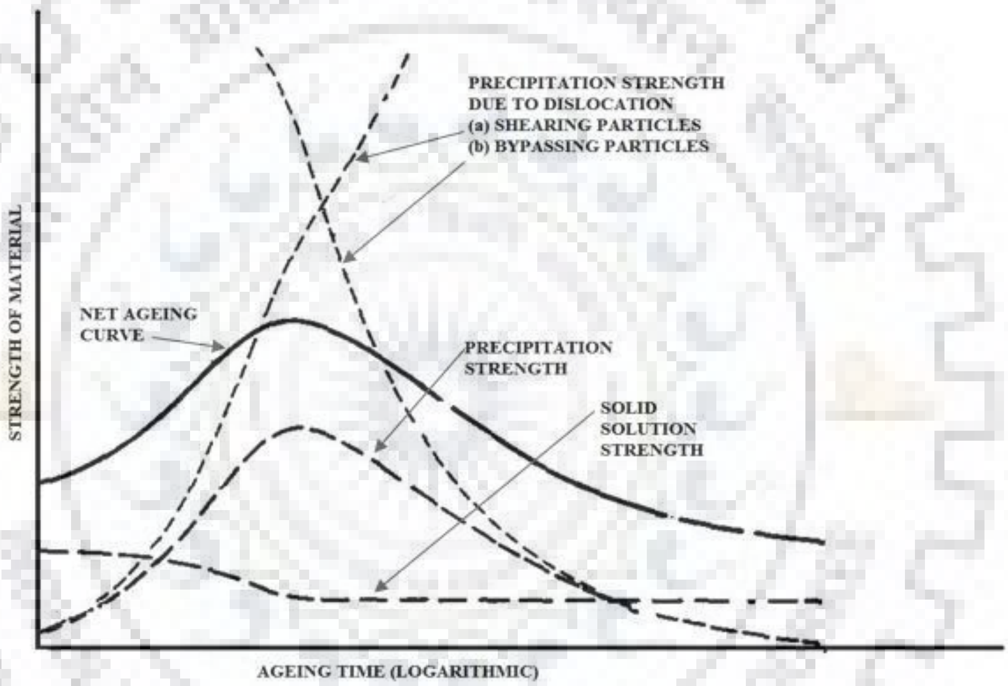


Fig 2.8 The shearing and by-pass mechanism during age hardening of the alloys.

2.4 METALLOGRAPHY

Metal and alloys are composed of crystals (grains). The configuration of the grains not only tell about the material property but also gives a idea about the constituent present in it and the way by which the alloys has produced. Apart from this a lot of information can be find out like presence of voids, cracks, grains orientation, grain sizes, inclusion in the alloys.

In this work, besides the mechanical behaviour of the AA 2014, the microstructure behaviour are also studied a bit using the optical instrument, scanning electron microscope and X-ray diffraction.

2.4.1 Optical Microscopy

For the microstructure, a specimen of dimension 1.5 cm x 1 cm x .6 cm is extracted from the plate.

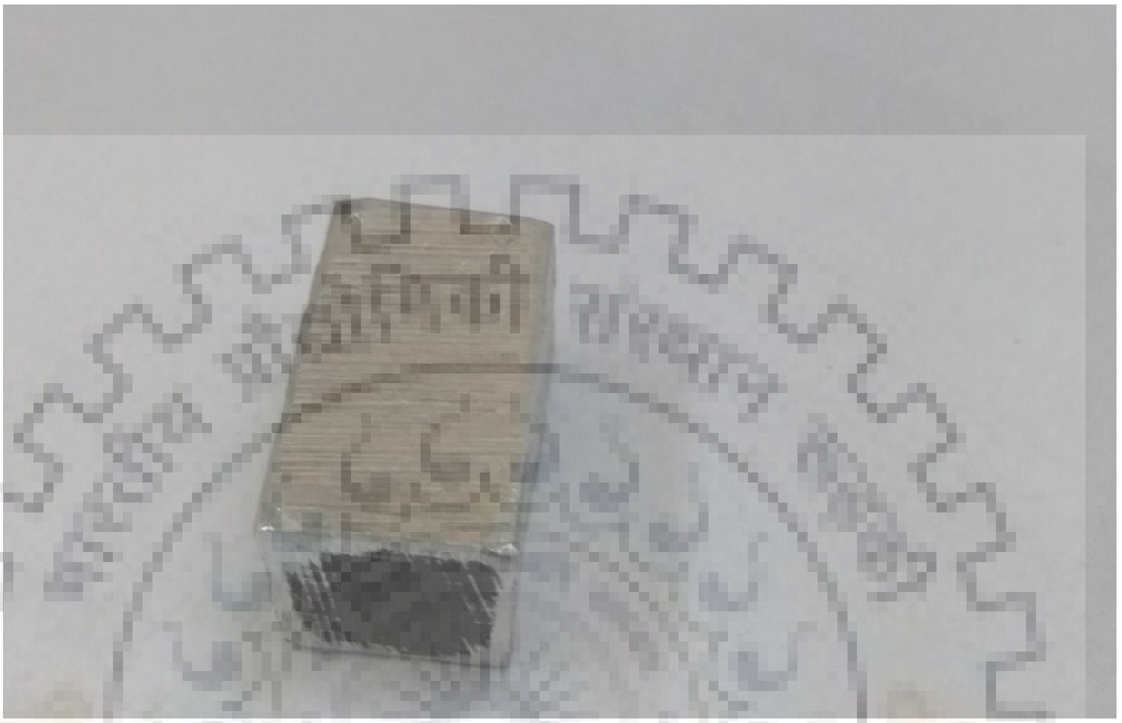


Fig 2.9 A Specimen selected for the purpose of the microstructure study.

The microstructure surface and the cross-section plane for the study of solution treated sample are shown in the diagram above. Micro-structure study is a direction dependent study. The direction is selected according to the need of the property to be evaluated. For the solution treated sample the surface of micro-structure is taken perpendicular to the longitudinal direction.

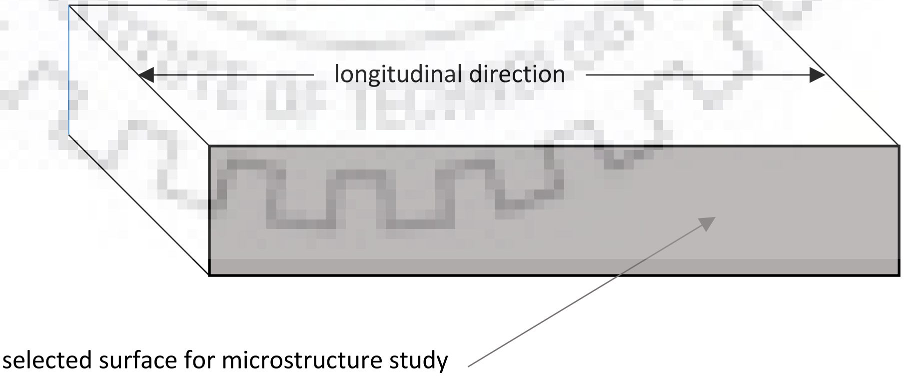


Fig 2.10 Orientation of the microstructure sample to the material.
A sample is selected shown with the face lateral to the longitudinal surface.

The preparation of microscopic specimen consists of following steps

(1) A described dimension of specimen is cut by means of hacksaw blade cutting machine with proper cooling. The effect of other parameter like heat generation in cutting, residual stress developed in cutting was minimized as much as possible.

(2) Grinding the small samples was very difficult, getting the flat surface for the microstructure and hardness was more difficult. Hence the sample were mounted in epoxy. A cold mounting is preferred so that any microstructure change does not takes place. Mounting the surface not only hold the sample easily but also help in keeping the sample optically flat.

(3) A number of emery paper has been used to grind the surface of sample. Starting from 220 grit size to remove the major surface ir-regularities, grinding end with 3000 grit sizes to obtain a surface finishing up to micron level. The coolant used in this process is running water through out. A running water not only cool the material but also remove the abrasive generated during grinding and keep the material from further corroding and grinding. A rotation to the specimen was also provided to eliminates the scratches created during grinding with every successive grinding paper change.

(4) Finishing the grinding with the 3000-grit emery paper gives the sample a very good surface but it is not sufficient for an optical microscope. The specimen was further polished with 6-micron magnesium-oxide power on a rotating wet cloth for at least half hour subsequently it is polished with 1-micron magnesium-oxide powder for several minutes until all the scratches are gone.

(5) The polished specimen further needs to etch for grain sizes, grain structure, their orientation and other details. The etchant used for this purpose are Keller's reagent. The chemical present are distilled water 90mL, Nitric acid 5mL, HF acid 2mL, HCl acid 3mL. The etchant attack on the grain boundary and make them visible in optical. A freshly prepared Keller's reagent is applied to the sample for not more than 15 second.

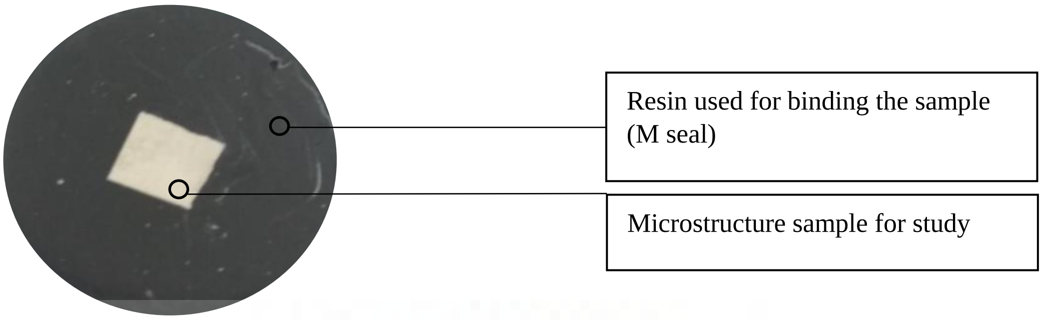


Fig 2.11 Arrangement of the sample for the microstructure study.
It helps the sample to remains optically flat and make it easy to handle.

Fig 2.12 Leica optical microscope used for the microstructure study.

2.4.2 Scanning Electron Microscope

Apart from optical microscope a FESEM devices are also used to investigate the microstructure of the material. FESEM devices are based on electron diffraction and gives highly clear and focused images. The sample preparation process for FESEM is same as the optical microscope sample, However the samples are finished with ½ micron magnesium oxide powders.

Fig 2.13 Scanning Electron microscope used for the study of the microstructure of the sample.

2.4.3 X-ray diffraction

x-ray diffraction is also performed to know the different chemical and precipitate present in the material during ageing. The XRD sample are cubic shape having a dimension of 10 mm x 0.5 mm x 03 mm. The sample are cleaned very carefully such that there remains no sign of any other impurities or other material present.

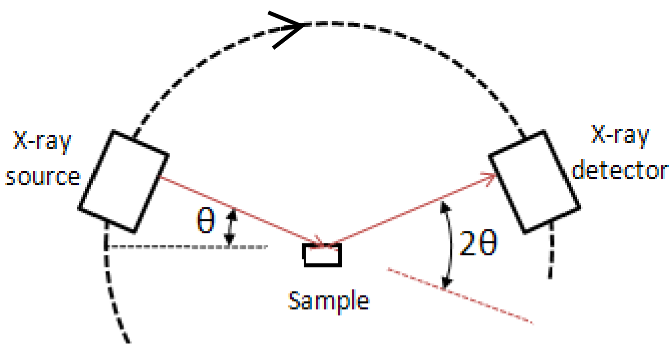


Fig 2.14(a) X-ray diffraction principal used for identifying particles.

Fig 2.14 X-Ray Diffraction instrument used for the XRD analysis of the sample.

2.5 Mechanical Testing Procedure

Various mechanical tests are performed to investigate effect of ageing behaviour on Aluminium Alloy 2014. A brief description of the sample preparation, various assumption and the test procedure are described here.

2.5.1 Hardness Test

Vickers's micro hardness is performed to know the effect of ageing on the material. A sample of dimension 10 mm x 10 mm x 8 mm are extracted from the material for hardness test. The sample are surface finished with emery papers to remove any scratches present and to clearly reveals the different diameter of the Vicker indenter. Vicker indenter are square in cross-section and are made of diamond.

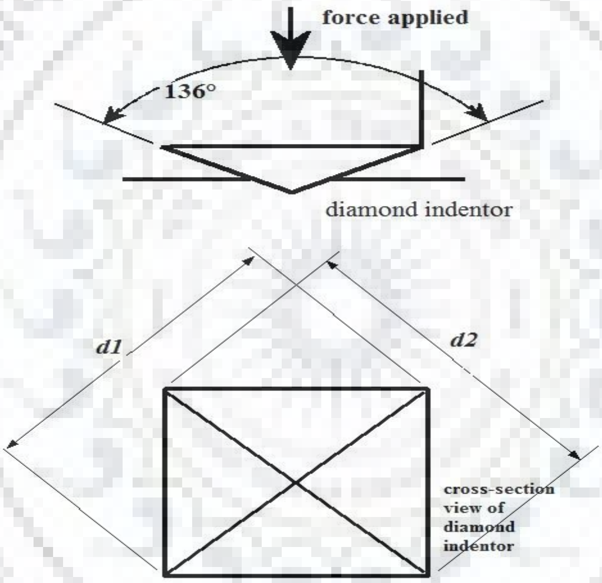


Fig 2.15 Vicker indenter cross-section view. the two diameters.

$$d = \frac{d1+d2}{2}$$; average diameter

$$HV = \frac{test\ force}{surface\ of\ indentation} \text{X}\ constant$$

$$HV = \frac{2F\sin\frac{\theta}{2}}{d^2}$$; θ = 136° indenter angle

2.5.2 Tension Test

A tension test represents probably the most important characteristic of a material mechanical properties. A stress strain curve obtained basically by fixing one end of the specimen and subjecting the other end to a controlled displacement. A load cell present at any end may calculate the amount of load acting with respect to the displacement.

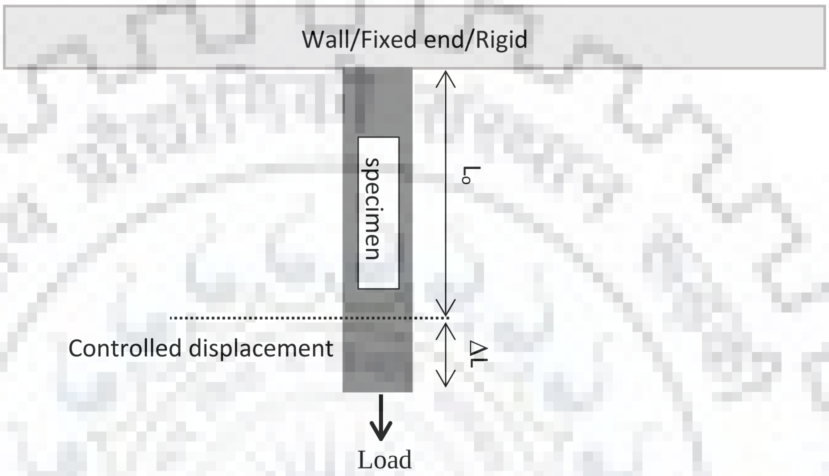


Fig 2.16 A general test setup for the Tensile test.

There are two types of Tension Testing Machine, one in which displacement is controlled and load is measured as a function of displacement. In the second type the load is controlled and the displacement is calculated as the function of load. Conventionally all the tension testing machine are displacement controlled. Few modern testing machines are load controlled.

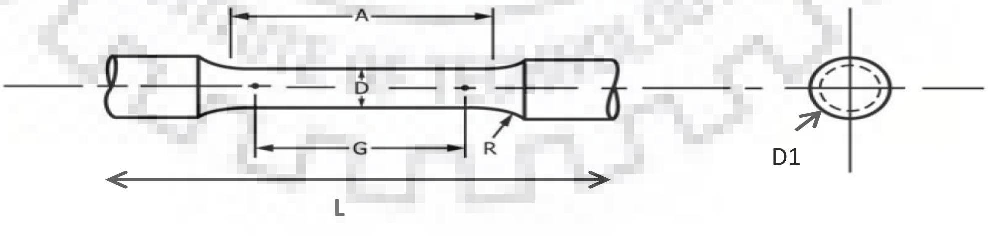


Fig 2.17 Tensile test specimen used for the tension test.

Table 2.2 Various dimension of tensile test specimen in mm.

Indicated letter	Dimension in mm	Remark / name
G	16	Gauge length
D	4	Gauge diameter
R	2	Radius of curvature
L	50	Total specimen length
D1	8	Grip diameter

A displacement and the corresponding load value automatically generated from the Tension Testing Machine as below. Further the stress-strain curve from the table can be generated from the table as follows.

Table 2.3 A result of displacement vs load obtained from UTM.

Sr No	Displacement (mm)	Load (kN)
1	..	..
2	..	..
3	..	..
..	..	..

$$strain = \frac{change\_in\_length}{original\_length}$$

$$stress = \frac{load}{Original\_Area}$$

Table 2.4 Conversion of load vs displacement into stress vs strain.

Sr No	Strain	Stress
1	..	..
2	..	..
3	..	..
..	..	..

A stress-strain plot obtained in general for material like aluminium, copper, Magnesium rather than steel is as follows.

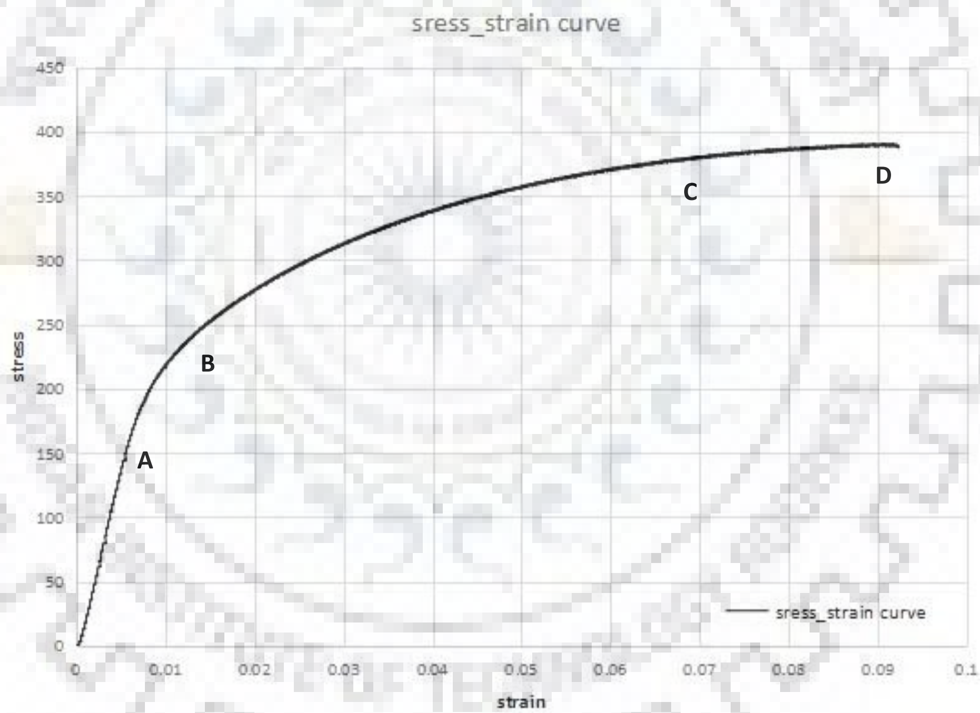


Fig 2.18 A general stress-strain plot obtained from the Tensile test.

Till point A the curve shows a linear relationship *ie* follows the Hooke's law. The slope of the line is known as young's modulus of elasticity. Portion between b and c are known as strain hardening region. The point D indicates the ultimate tensile strength of the material. A line parallel to line OA and having offset of 0.2% is drawn for finding out the yield tensile strength.

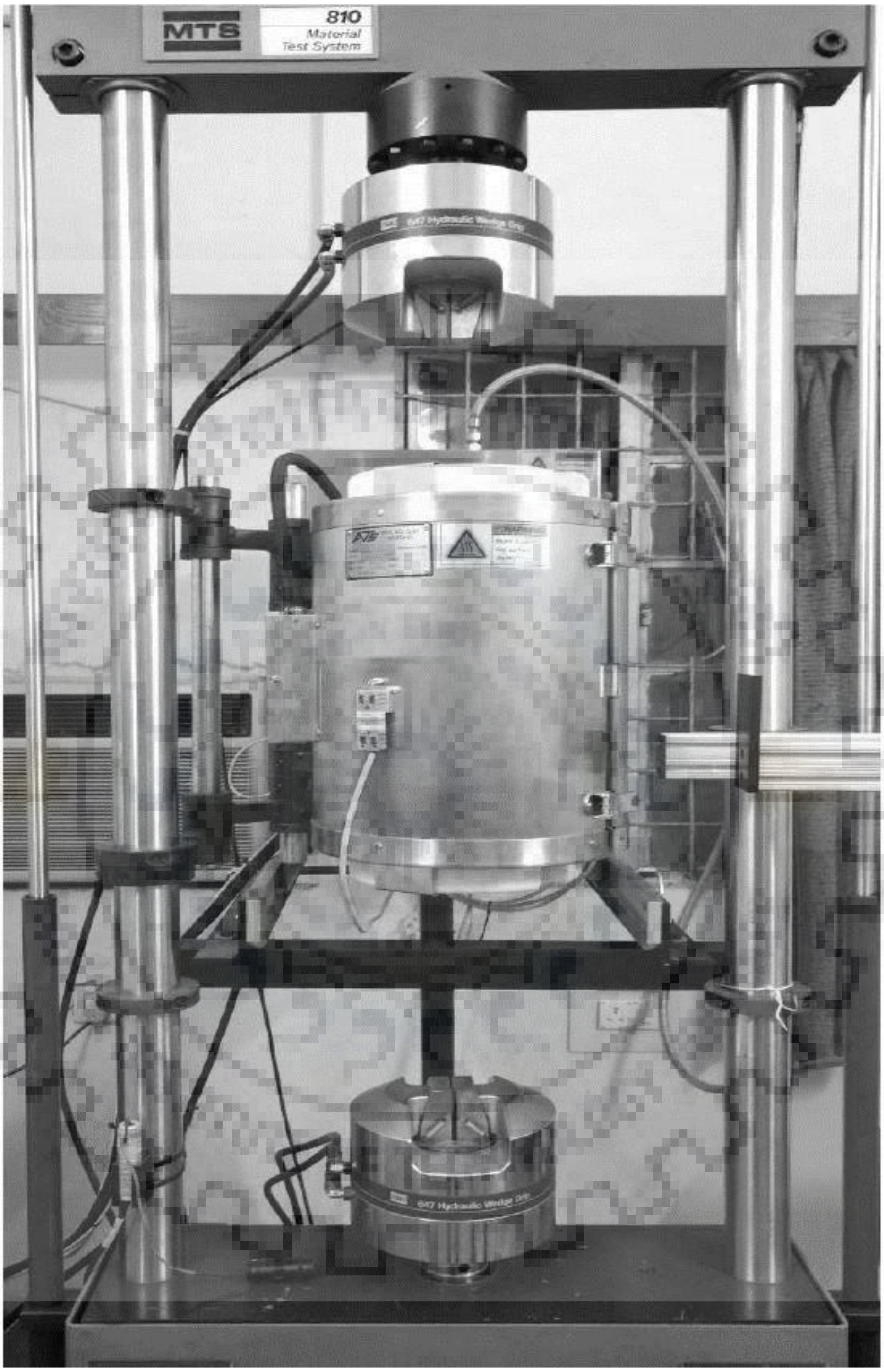


Fig 2.19 Universal Tensile Testing machine used for the tension test.

2.5.3 Fatigue Crack Growth

There are numerous ways of material failure and the type of failure depend of the type of loading, material characteristics, environmental conditions etc. fatigue crack growth is also among them. FCG characteristics become quite important for the component having long service and under dynamics loading condition. Fatigue are catastrophic failure. Dynamics load circumstances lead the material failure condition quite below as compared to static loading failure criteria. Researches in this field shows crack propagation as the reason. *ie* every dynamic cycle of load propagates the crack a little bit. The phenomenon follows till a critical crack length. The critical crack length is the value at which the material fails. 2014 Aluminium alloys are broadly used in aerospace field. The aerospace materials sustain various types of loads like Bending, Torsion, Fatigue, Tension and compression, Thermal etc. Concerning these dynamics loads, Fatigue Characteristics of these alloys become important to understand while designing the component. In this dissertation report the Fatigue Crack Growth characteristics are co-related with the ageing phenomenon. Compact specimen according to ASTM-E647 has been made for FCG test. The specimen is shown below.

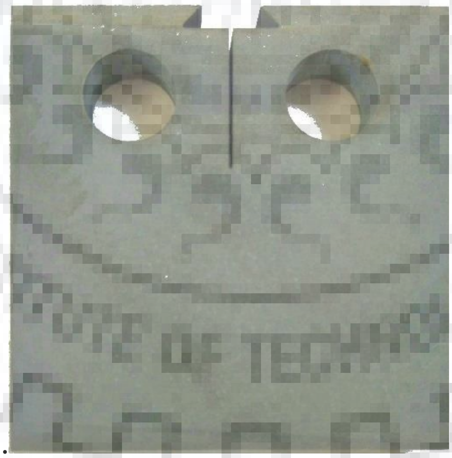


Fig 2.20 CT specimen diagram for FCG testing.

The standard constant W is taken as 32mm. Rest of the dimension are in the proportion as mentioned in ASTM-E647.

Notches in the samples were formed by Wire Drawing Machine having wire of diameter 0.4mm. Making of specimen follows the same procedure as the tensile specimen *ie* ageing followed by solution treatment. A special setup was arranged for FCG test. The loads are applied in the specimen are through the cylindrical pin which are themselves hinged in a mechanical setup. The diagram illustrating the setup are shown below.

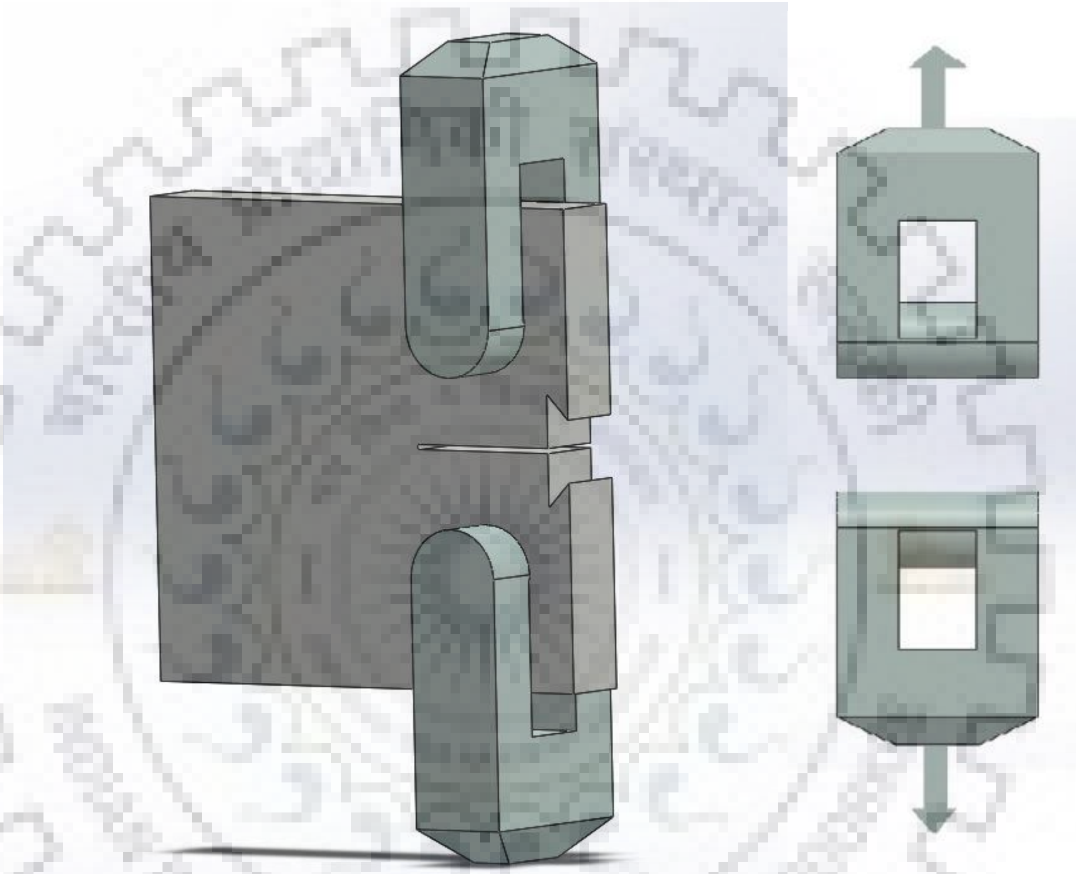


Fig 2.21 FCG specimen arrangement for testing

The edge notches are constructed for the measurement of the crack tip opening displacement. It is used for fitting extensometer. An MTS extensometer (MODEL 632.020-20) is used to measure the crack tip opening displacement as shown in the fig 2.22. The sample inclination or tilt angle are checked properly with spirit level.

Fig 2.22 FCG specimen arrangement in the MTS 810 machine.

Chapter 3

MECHANICAL BEHAVIOR AND CHARACTERIZATION

3.1 INTRODUCTION

The lightweight Aluminium attracted the attention of engineers heavily. However, its low strength does not allow it for industrial purpose. Since last few years engineers has tried and developed a lot of mechanical and thermal and chemical processes to enhance the mechanical properties of Aluminium Alloy. Alloying of the Aluminium by adding some additional element for increasing the strength and various properties are one of them. There are also many mechanical and thermal processes through which Aluminium properties can be improved. SPD technique like Cryo-Rolling, Equal Channel Angular Processing (ECAP), High Pressure Torsion (HPT), Repetitive Corrugation Method are mechanical ways to improve properties of Aluminium Alloy, whereas heat treatment like annealing, aging, solution treatment are thermal processes to improve mechanical properties. In this work aging is selected as the method for improving the mechanical properties of Aluminium Alloys 2014. The formation of precipitate during ageing treatment are responsible for increase in the strength. The precipitates act like restriction in the way of dislocation movement and thus result in the increase in the yield strength and ultimate strength of the material.

This chapter deal with the microstructure characterization and mechanical behaviour of the solution treated and aged sample for different time and temperature. Tension testing, hardness testing, impact testing was performed to find the effect of solution treatment and aging on the Aluminium Alloys.

3.2 MICROSTRUCTURE CHARACTERIZATION

Besides the study of mechanical properties, microstructure characteristics has also been studied using few techniques such as optical microscope, field emission scanning electron microscope, electron back scattered diffraction, X-ray diffraction.

3.2.1 Optical Microscopy

A specimen of dimension 10 mm x10 mm x 5 mm are prepared according to the ASTM standard. The grinding of the sample has been done with silicon emery paper starting with 220 grit sizes and up to 3000 grit sizes. The intermediate grit sizes are 400, 600, 800, 1000, 1200, 1500 and 2000. A Keller's etchant is used for 15 second to etch the sample after grinding. Microstructure has been analyzed using optical microscope of Solution treated and different aged sample.

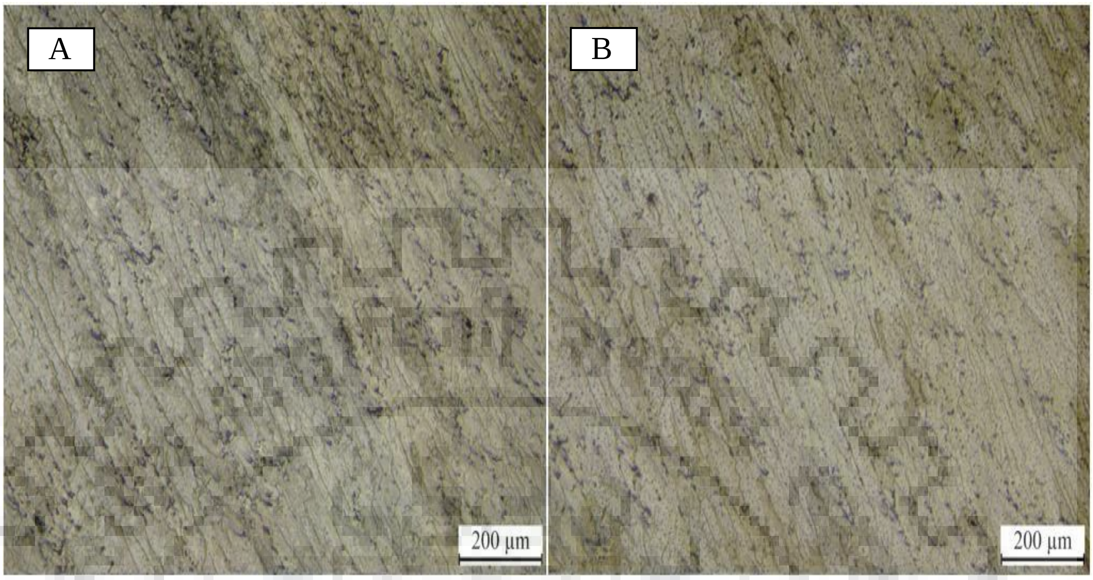


Fig 3.1 Optical microstructure of solution treated Aluminium Alloy 2014 T651; (A) the optical microstructure obtained of as received material; (B) optical microscope obtained after solution treatment.

Fig 3.1 shows the microstructure of the before and after solution treated sample at 504°C for 2Hour. The alloys microstructure reveals elongated grains along with the very little amount of precipitate. The images reveal the elimination of second phase particles formed at the grain boundary in the as received material.

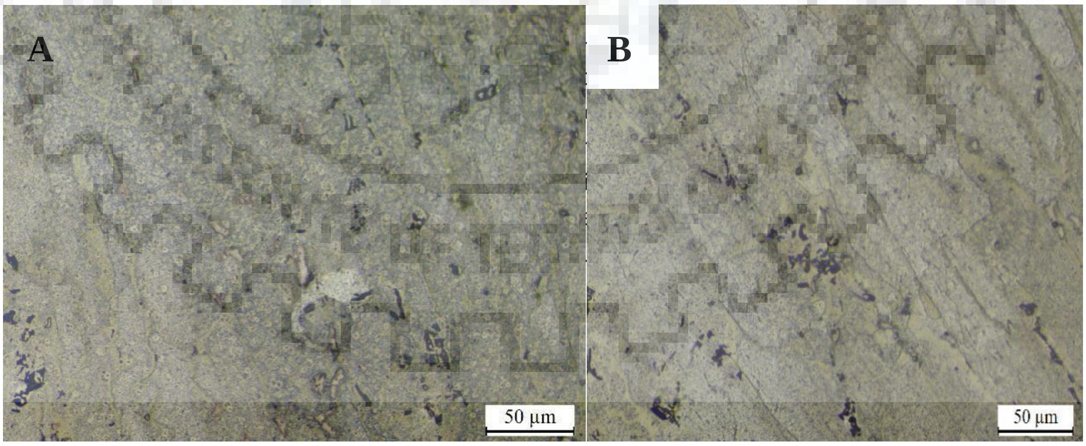


Fig 3.2 Optical microstructure of sample aged at 160°C for; (A) 4Hour; (B) 10Hour of solution treated Aluminium Alloy 2014 T651.

Figure 3.2 are the optically microscope images of the aged sample for different time period at 160°C. figure A describe the image obtained after ageing the sample for 4Hour. The black particle shown in the images are what should be Al_2Cu or Mg_2Cu.

The image reveals that there is very less amount of precipitate present in figure 3.2(A). The figure 3.2(B) are the microstructure of the sample aged at 160°C for 10Hour. The figure reveals that there is more amount of precipitates present in the microstructure as compared to the 4-hour ageing.

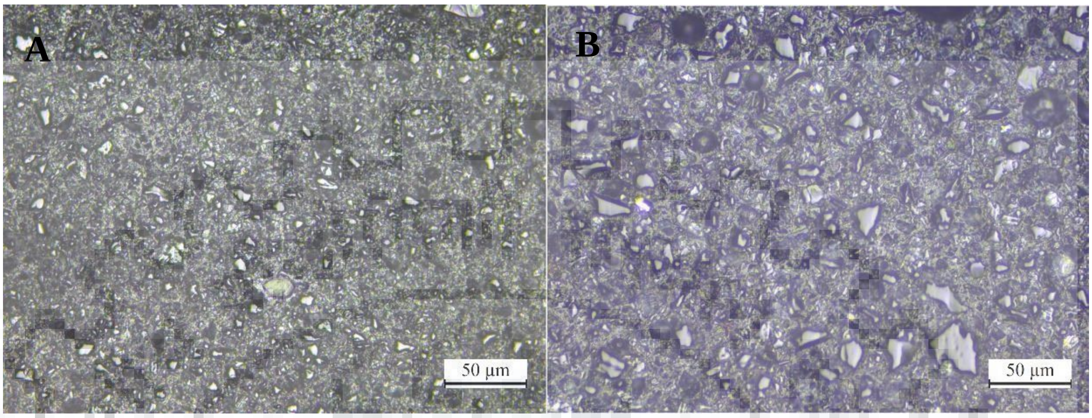


Fig 3.3 Optical microstructure of sample aged at 190°C for; (A) 4Hour; (B) 10Hour of solution treated Aluminium Alloy 2014 T651.

Figure 3.3 shows the microstructure obtained after Ageing the sample at 190°C for different hour. Figure 3.3(A) describe the image obtained after Ageing the sample for 4Hour. The precipitates particles which are clearly shown in the figure are of less sizes than figure 3.3(B). the figure 3.3(B) showing the images of the sample aged for 10Hour at the temperature of 190°C.

3.2.2 Field Emission Scanning Electron Microscope

A specimen of same size and shape as optical specimen is selected for SEM analysis. Similar procedure has been followed for SEM sample preparation. SEM images are highly magnified images giving a clear material structure.

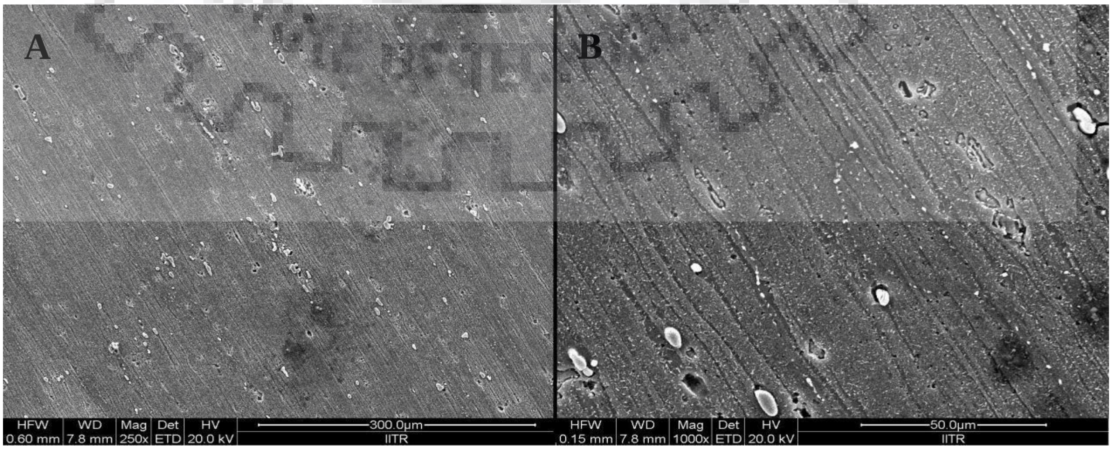


Fig 3.4 FESEM image of ST sample; (A) a resolution of 300µm; (B) a resolution of 50µm.

Fig 3.5 showing the SEM images of solution treatment sample. The images reveal that there is very less amount of precipitates present in the sample. The white particle present in the figure are what should be Al_2Cu or Mg_2Si precipitates and the surrounding area should be α-Al based solid solution.

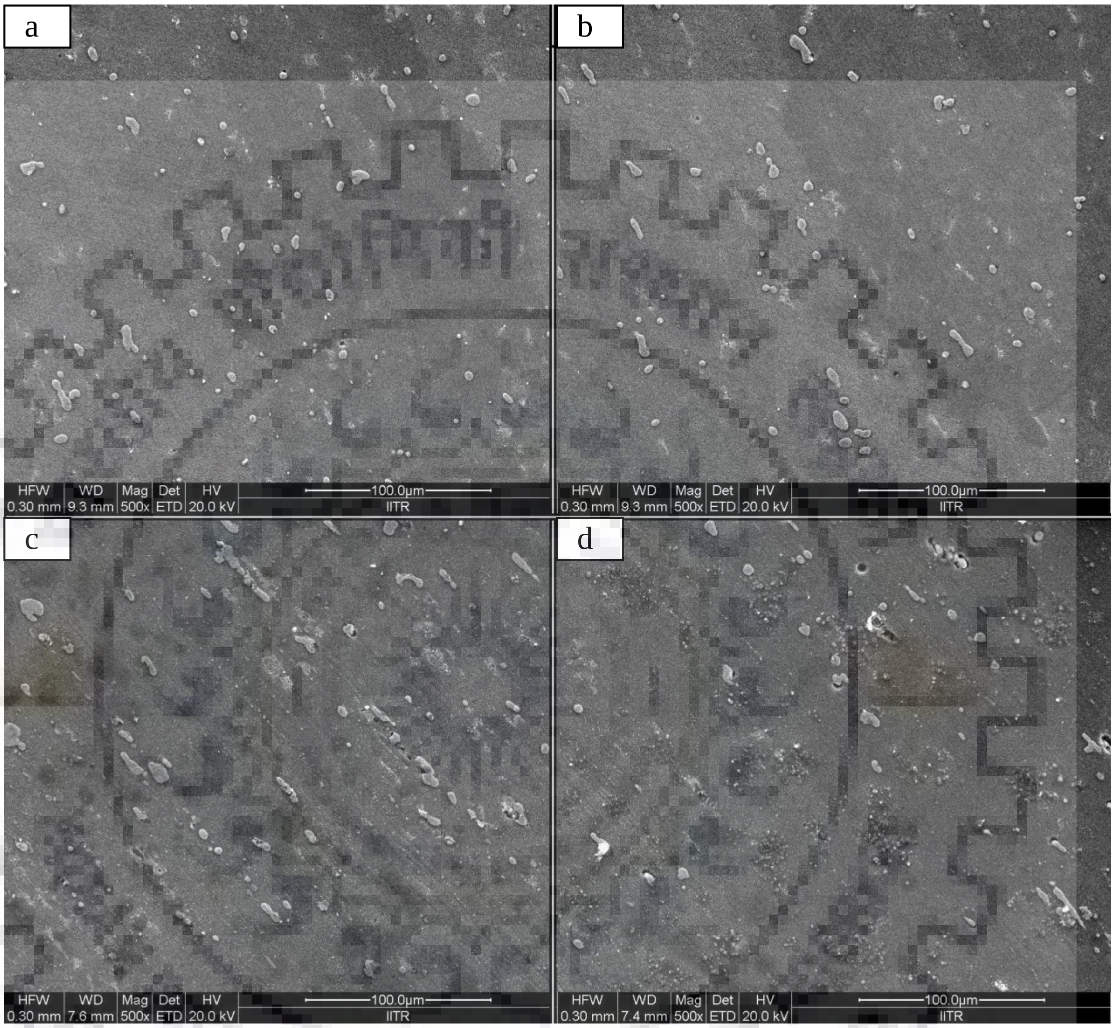


Fig 3.5 SEM images of aged sample at 190°C; (a) sample aged at 190°C for 4H; (b) sample aged at 190°C for 6H; (c) sample aged at 190°C for 8H; (d) sample aged at 190°C for 10H).

Fig 3.5 Shows the microstructure of aged sample for different time period obtained by SEM. Result reveals that the solution treated sample possess very little amount of $CuAl_2$ precipitate. The microstructure of aged sample in figure 3.6 reveals some more amount of $CuAl_2$ precipitate in comparison to ST sample. The formation of precipitate follows the sequence SSSS (super saturated solid solution). *ie* the solution treatment process with quenching locked the solid solution in super saturated state. The precipitation process starts with development of nano-meter size particles known as Guinier-Preston zones. These Guinier-preston particles are coherent in nature and are optically invisible. GP zones are initial form of precipitate. The particle in white circles should be $CuAl_2$ or Mg_2Si precipitate fully grown. The SEM images at 190°C shows some of the different particles also. The particles in the white region are showing some of other precipitates developed in the material. The newly developed precipitates increase the amount of present precipitates. This decreases the material properties as shown in the hardness and tension test result.

3.2.3 X-RAY DIFFRACTION

Apart from the microstructure study XRD studies are also conducted to find out the presence of different precipitates at different ageing condition.

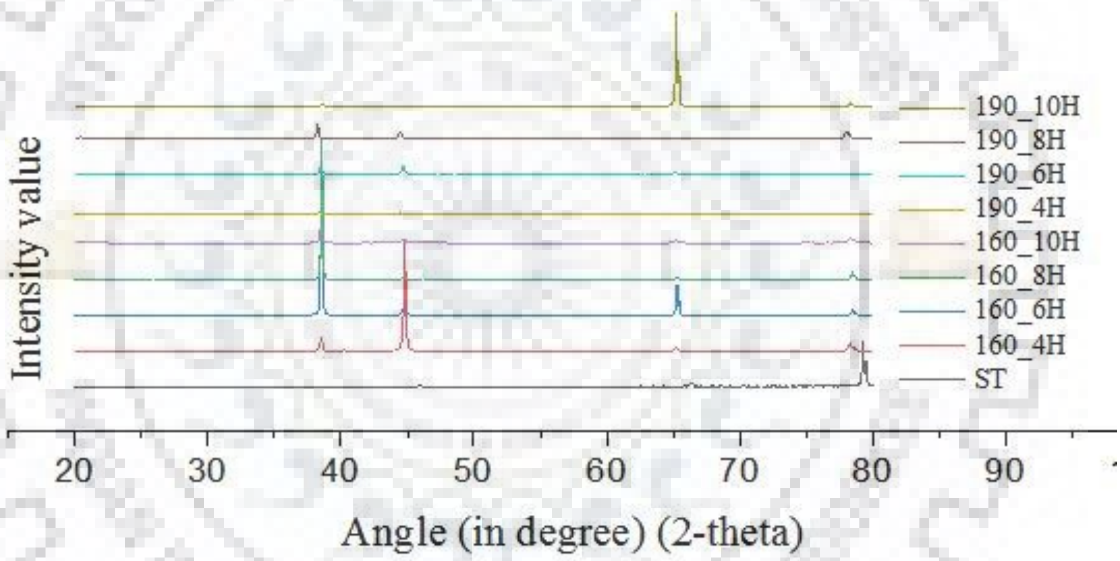


Fig 3.6 XRD plot for different condition. The sample are named according to their state.

Figure 3.6 indicate the XRD plot obtained for different condition. XRD plot for solution treatment shows a little presence of $CuAl_2$ and $MgSi_2$ along with AlFeSi. The quantity of $CuAl_2$ is very less in case of solution treatment sample however, it is quite more in case of aged sample. Appearances of some others particles $Al_2Si_6Mg_8Cu_2$, and $Al_{15}Si_2$ in little amount after precipitation. The different peak obtained was at 38.4747, 44.73,65.0958 and 78.177° respectively.

3.3 Mechanical behaviour

Various mechanical test has been performed to investigate the effect the ageing on the Aluminium Alloy 2014. A detailed analysis of each test and the results obtained from them are described here.

3.3.1 Micro Hardness Testing

Vicker hardness test are performed to know the effect of ageing on Aluminium Alloy 2014. the samples preparations are according to ASTM E92-17, ASTM E3-11 (standard test method for Vicker hardness, standard procedure for metallography sample preparation). Minimum of 6 sample are averaged for remove any error in hardness value over different location.

Table 3.1 Hardness value over different Solution treatment time period.

Sr No	Specimen condition	Hardness value (HV)
		Surface I
1.	Solution treated 504°C_2H	107.77
2.	Solution treated 504°C_2.5H	105.98
3.	Solution treated 504°C_3H	104.25

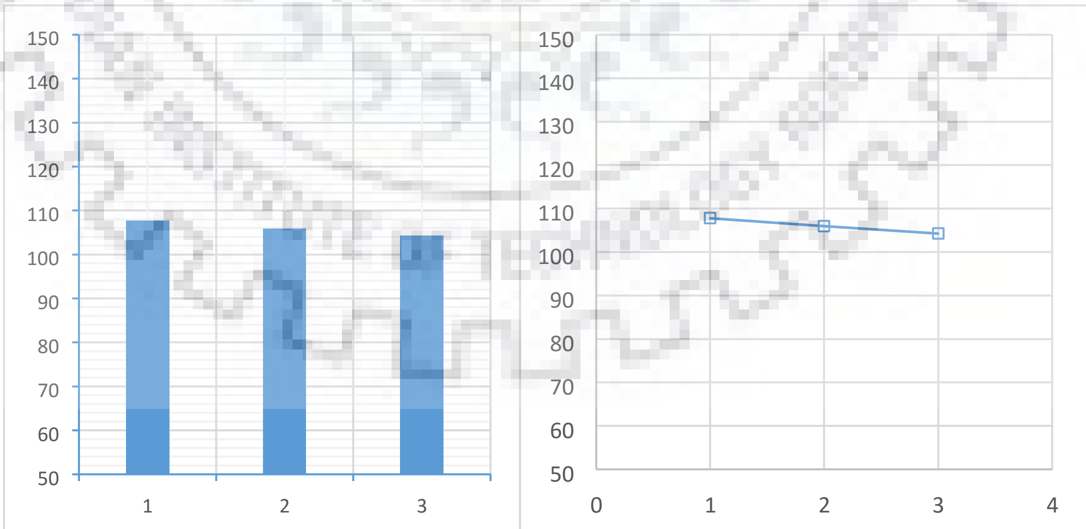


Fig 3.7 Static plot for hardness variation over different solution treatment time, (1) stand for 2Hour; (2) for 2.5Hour; (3) for 3Hour.

Table 3.2 Hardness value over Ageing at 190°C for different time period.

Sr No	Specimen condition	Hardness value (HV)
		Surface I
1.	Solution treatment 504°C_2H	107.77
2.	Aged at 190°C for 4H	119.98
3.	Aged at 190°C for 6H	123.11
4.	Aged at 190°C for 8H	125.23
5.	Aged at 190°C for 10H	120.74

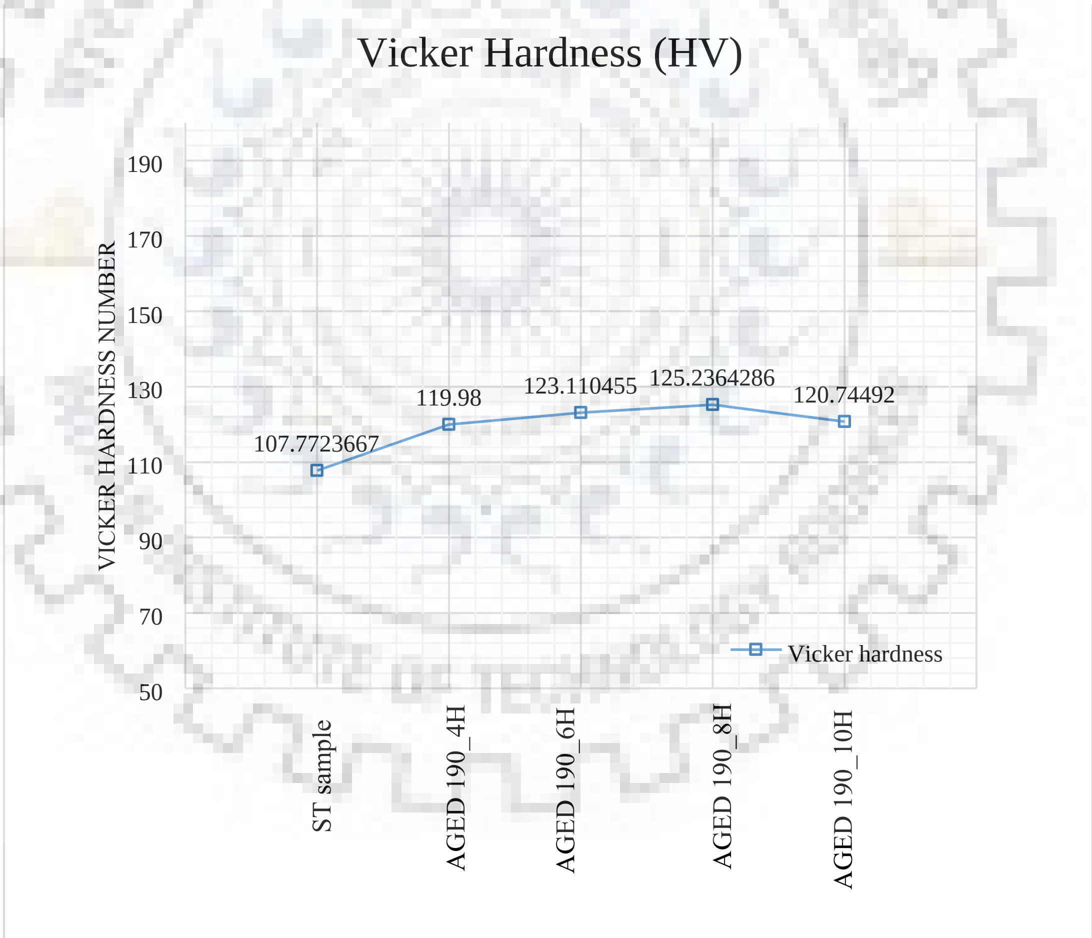


Fig 3.8 Hardness value obtained over different time period of ageing at 190°C.

Table 3.3 Hardness value over Ageing at 160°C for different time period.

Sr No	Specimen condition	Hardness value (HV)
		Surface I
1.	Solution treatment 504°C_2H	107.77
2.	Aged at 160°C for 4H	116.59
3.	Aged at 160°C for 6H	121.732
4.	Aged at 160°C for 8H	136.76
5.	Aged at 160°C for 10H	137.511

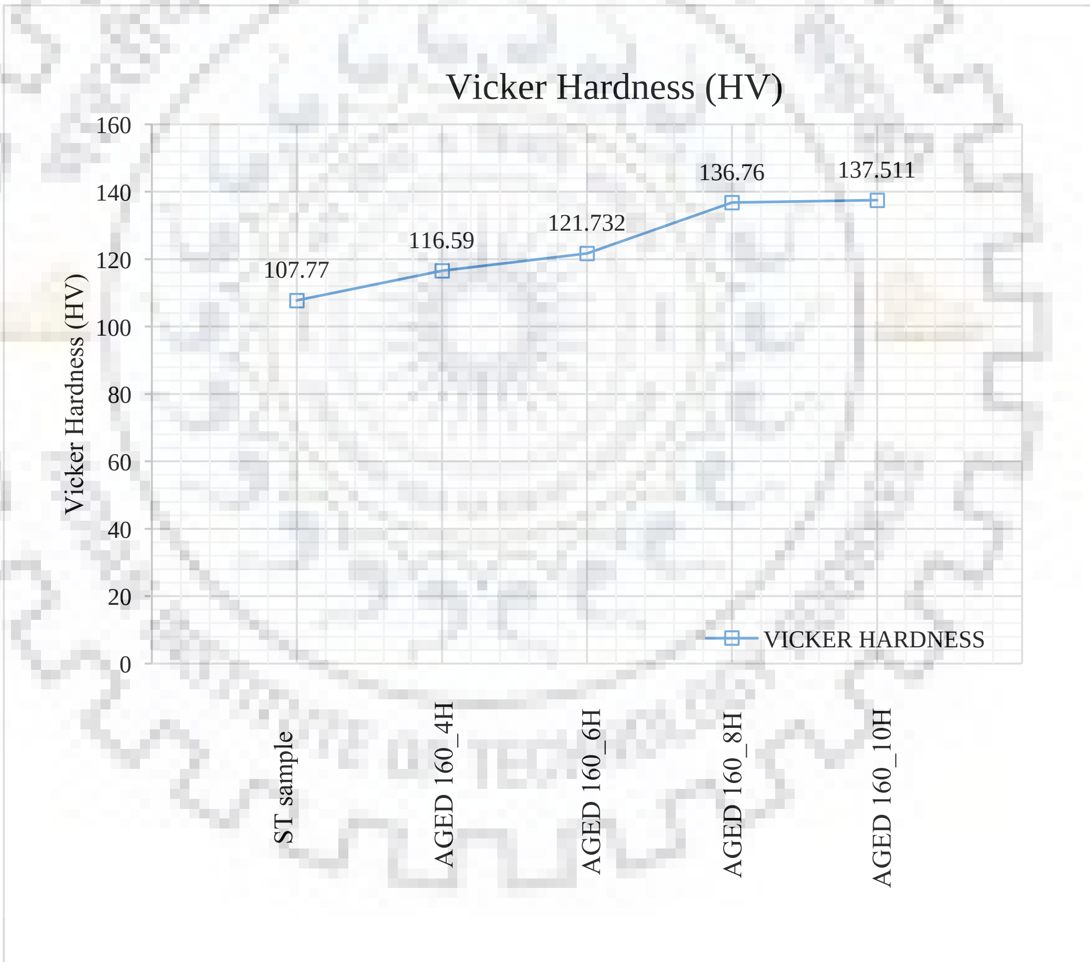


Fig 3.9 Hardness value obtained over different time period of ageing at 160°C.

Table 3.4 Hardness value over Ageing at 130°C for different time period.

Sr No	Specimen condition	Hardness value (HV)
		Surface I
6.	Solution treatment 504°C_2H	107.77
7.	Aged at 130°C for 4H	119.98
8.	Aged at 130°C for 6H	123.11
9.	Aged at 130°C for 8H	125.23
10.	Aged at 130°C for 10H	129.74

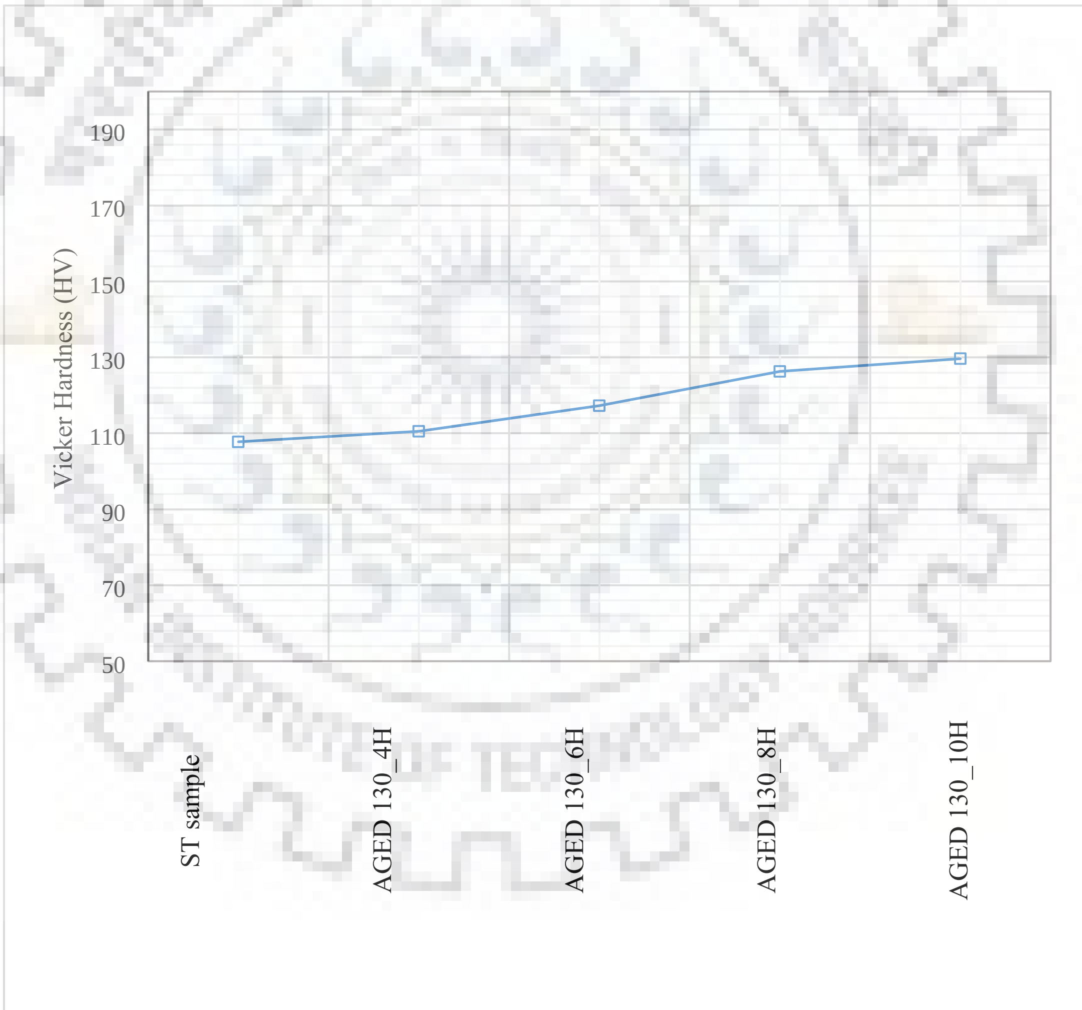


Fig 3.10 Hardness value obtained over different time (in hour) period of ageing at 130°C.

3.2.2 Tension Testing

tension testing is the most basic test and the necessary test. It gives the information regarding the ultimate strength of the material, yield strength of the material, ductility values, elongation in the material, young's modulus of the material and a lot of information. Tension testing consider a stress vs strain plot obtained via UTM machine through a controlled displacement module.

3.2.2.1 Solution Treatment effect

The samples are solution treated at 504°C for different time period to see the effect. The results are as follows

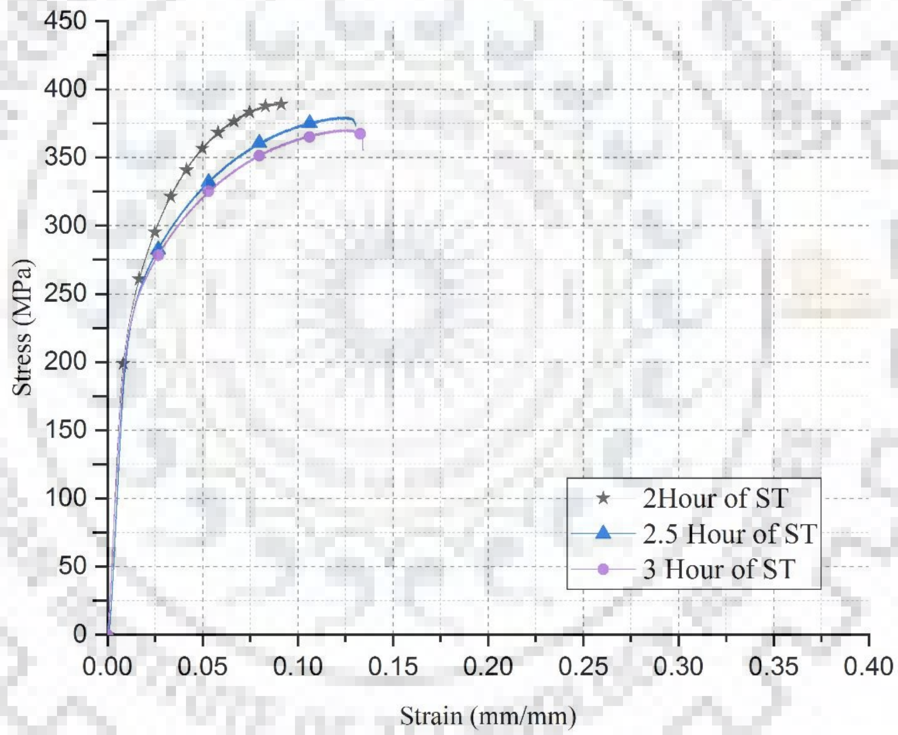


Fig 3.11 Stress Strain plot for different solution treatment time period.

Figure 3.11 reveals that the strength of the material decreases with increasing the time period of solution treatment. After solution treatment for 2Hour the UTS and YTS was found to be 390MPa and 240MPa which decreases to 379MPa and 220MPa after 2.30Hour treatment. The result shows further decrease in UTS and YTS with increasing time period of 3Hour. A corresponding increase obtained in %elongation with respect to solution treatment time period. The % elongation was found to be 14.24 during 2Hour treatment which increases to 16.4 and 20.23 corresponding to 2.3Hour and 3Hour respectively. The decrease obtained in the UTS can be understand on the basic of decrease in the precipitation amount during the formation of solid solution and homogenization process.

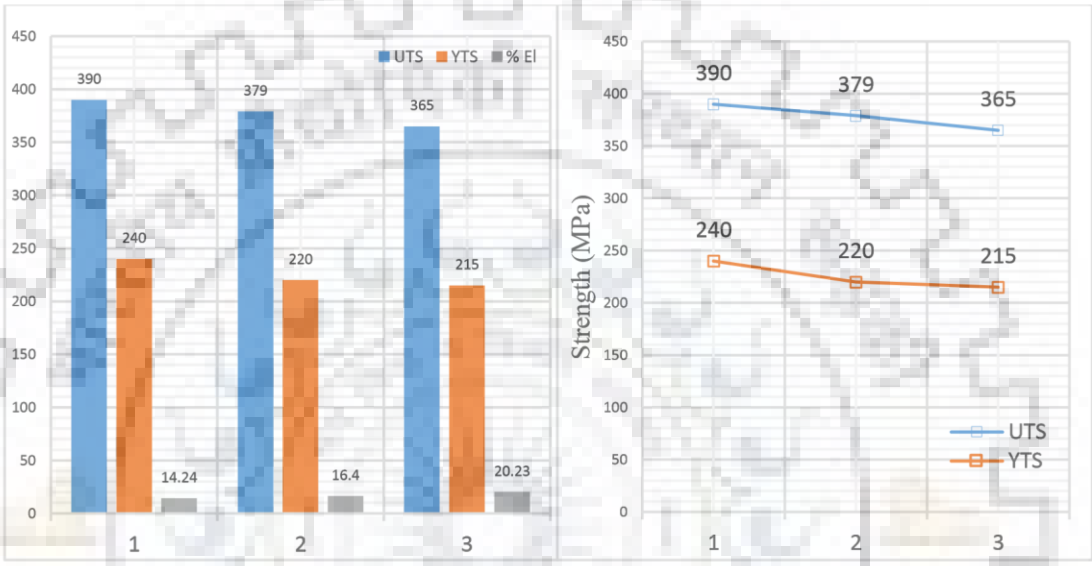


Fig 3.12 Statics plot for different solution treatment condition; (1) ST for 2H; (2) ST for 2.3H; (3) ST for 3H.

3.2.2.2 Ageing treatment

A tension test plot considering the ageing effect of Aluminium Alloy is as follows.

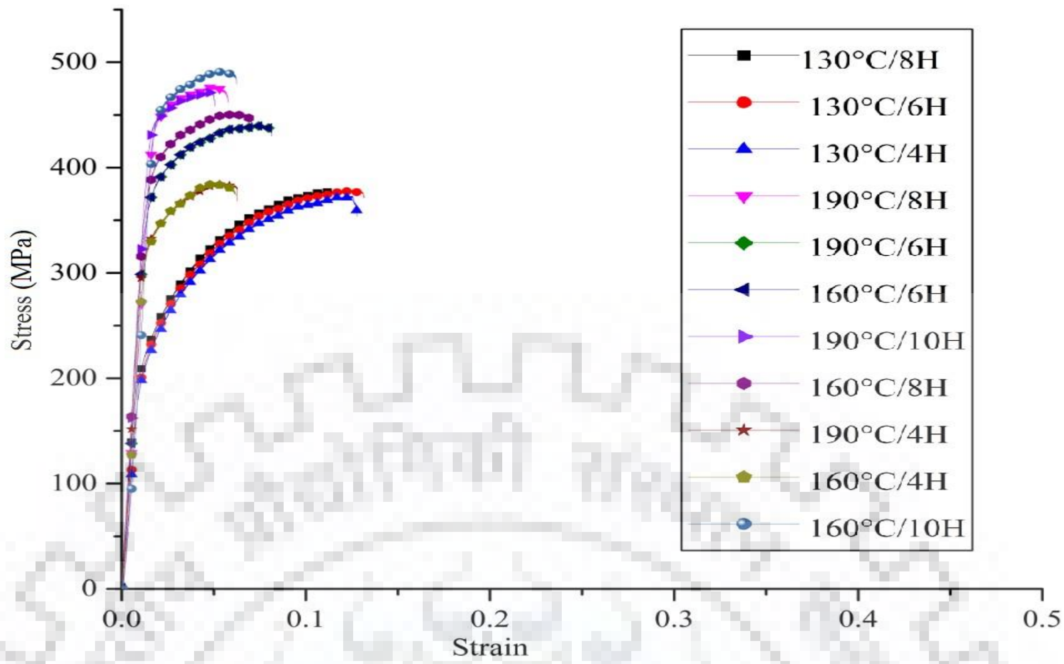


Fig 3.13 Stress Strain plot for different Aged temperature and time period.

Table 3.5 Different tensile data obtained for different Ageing treatment and different time period.

Sr No	Specimen	UTS (MPa)	YTS (MPa)	%Elongation
1.	ST 504°C _AGED 130°C_4H	360	220	12.78
2.	ST 504°C _AGED 130°C_6H	365	230	13.182
3.	ST 504°C _AGED 130°C_8H	375	245	11.57
4.	ST 504°C _AGED 160°C_4H	375	325	6.178
5.	ST 504°C _AGED 160°C_6H	440	375	8.13
6.	ST 504°C _AGED 160°C_8H	455	410	7.603
7.	ST 504°C _AGED 160°C_10H	490	440	6.231
8.	ST 504°C _AGED 190°C_4H	380	330	6.29
9.	ST 504°C _AGED 190°C_6H	440	380	8.13
10.	ST 504°C _AGED 190°C_8H	475	450	5.76
11.	ST 504°C _AGED 190°C_10H	465	450	5.042

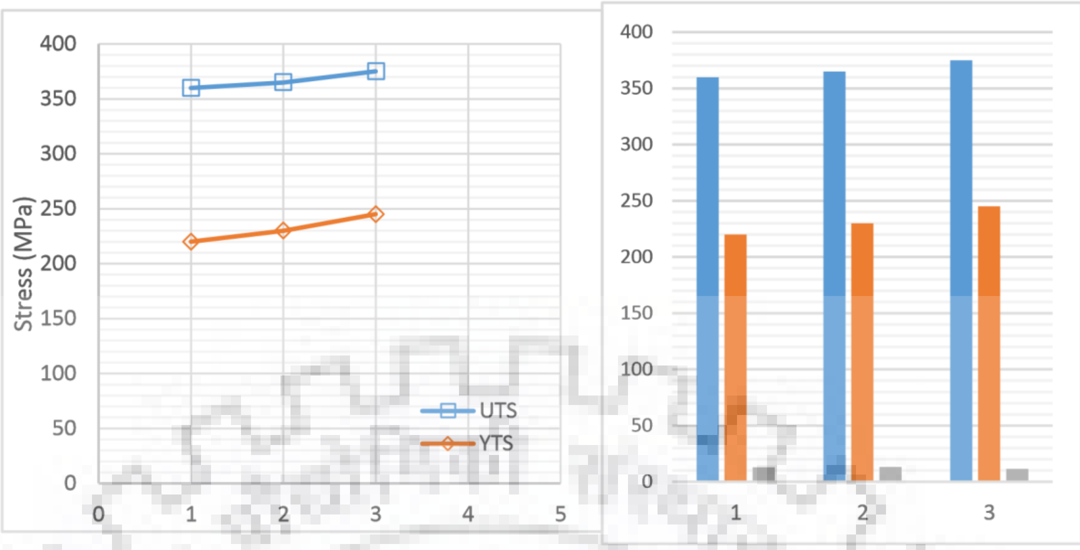


Fig 3.14 Statics plot for ageing at 130°C for different time period condition; (1) Ageing at 130°C for 4Hour; (2) Ageing at 130°C for 6Hour; (3) Ageing at 130°C for 8Hour.

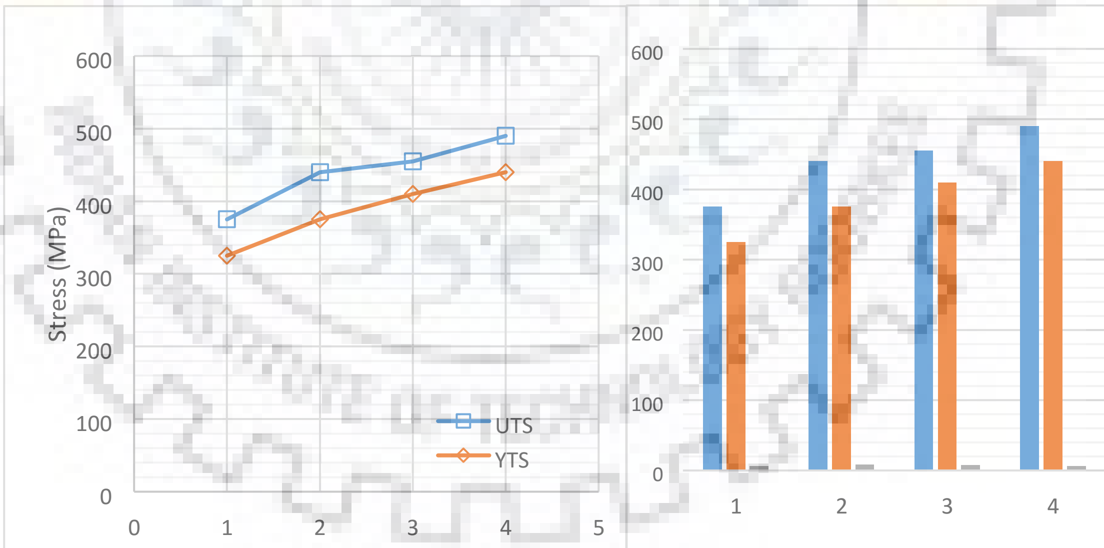


Fig 3.15 Statics plot for ageing at 160°C for different time period condition; (1) Ageing at 160°C for 4Hour; (2) Ageing at 160°C for 6Hour; (3) Ageing at 160°C for 8Hour.; (4) Ageing at 160°C for 10Hour.

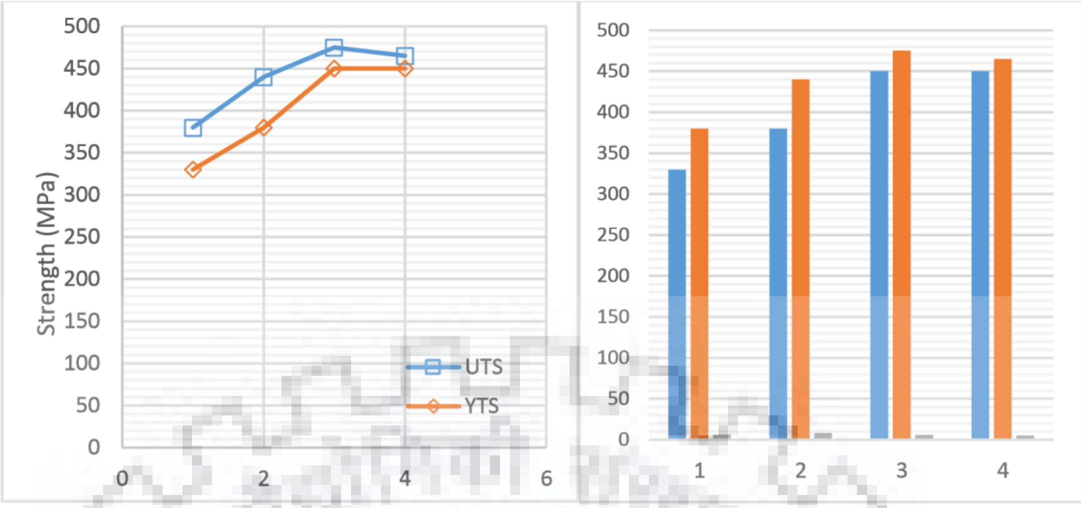


Fig 3.16 Statics plot for ageing at 190°C for different time period condition; (1) Ageing at 190°C for 4Hour; (2) Ageing at 190°C for 6Hour; (3) Ageing at 190°C for 8Hour.; (4) Ageing at 190°C for 10Hour.

Figure 3.12, 3.13 and 3.14 showing the result of ageing temperature and time over tensile properties of AA2014T6 solution treated sample. Figure 3.12 indicates the effect of ageing at 130°C for different time period. From the plot it is clear that the there is not much improvement in the tensile properties for ageing at 130°C. there is little amount of improvement if the time period of the ageing at 130°C increase. A significant improvement in result is obtained at ageing at 160 and 190. At ageing temperature of 160, initial UTS and YTS value are found to be 375 and 325MParespectively. With the increase in the time period of the ageing this value increased to 440 and 375, 455 and 410, and last 490 and 440MPa as UTS and YTS respectively. However, a simultaneous decrease in the value of % el obtained with the increasing the time period of ageing. The % el at 4 Hour of ageing found to be 8.13. It decreases to a value of 6.23 as a result of ageing for 10Hour. The result obtained for ageing at 190 shows an increase in the value of UTS and YTS up to 8 Hour of ageing and then it starts decreasing. At 4 Hour of ageing the UTS and YTS is found to be 380 and 330MPa. It increases to a value of 475 and 450MPa as UTS and YTS respectively up to a ageing time period of 8 Hour. For a time period of ageing at 10Hour the UTS and YTS are found to decrease as 465 and 450MPa was obtained as UTM and YTS values. The % el was found to be 6.29 for 4 Hour of ageing. It increases to a value of 8.13 for 6 Hour of time period. The % el was found to decrease with further increase in the time period and has a lowest value of 5.042 for a time period of 10 Hour of ageing.

3.2.3 Fatigue crack growth

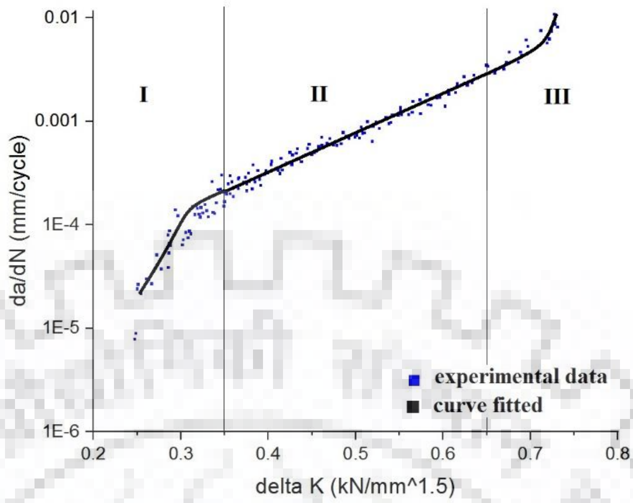


Fig 3.17 A typical FCG plot and its explanation.

As the fig 3.17 reveals, FCG plots are divided into three regions. Regions I contain the threshold value of the delta K below which there is no crack propagation at all. Fatigue crack growth in this region depend on microstructure. Size and orientation of grains quite play a role for the fatigue crack growth in this region. The II region follows Paris power law. Region II give the life of the sample subjecting to the fatigue loads. Region III is the case of final fracture. At this point the crack growth accelerate and final fracture occur.

$$\Delta K = K_{MAX} - K_{MIN}$$

$$R = \frac{K_{MIN}}{K_{MAX}}$$

$$\frac{da}{dN} = f(\Delta K, R)$$

FCG test were carried out by hydraulic MTS machine. The Plot 3.17 represent the fatigue crack characteristics for different ageing parameter.

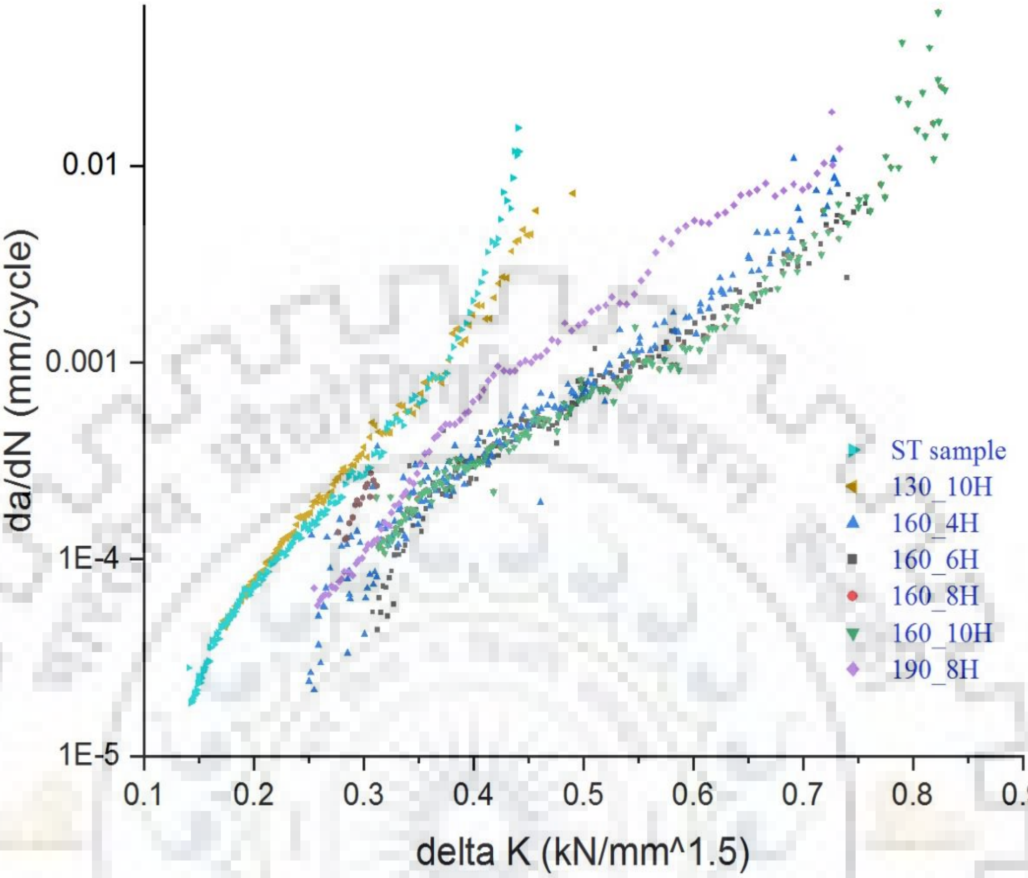


Fig 3.18 FCG plot obtained for solution treatment and different ageing parameter.

Figure 3.17 reveal an increase in crack resistance for aged sample as compared to the solution treatment sample. However, there is not sufficient difference obtained for ageing at 130°C ageing as the plot indicate. The result obtained are quite good for the aged sample for temp 160°C. they show a hike with increase in ageing time period. A max resistance against fatigue is obtained in case of 10 Hour of treatment.

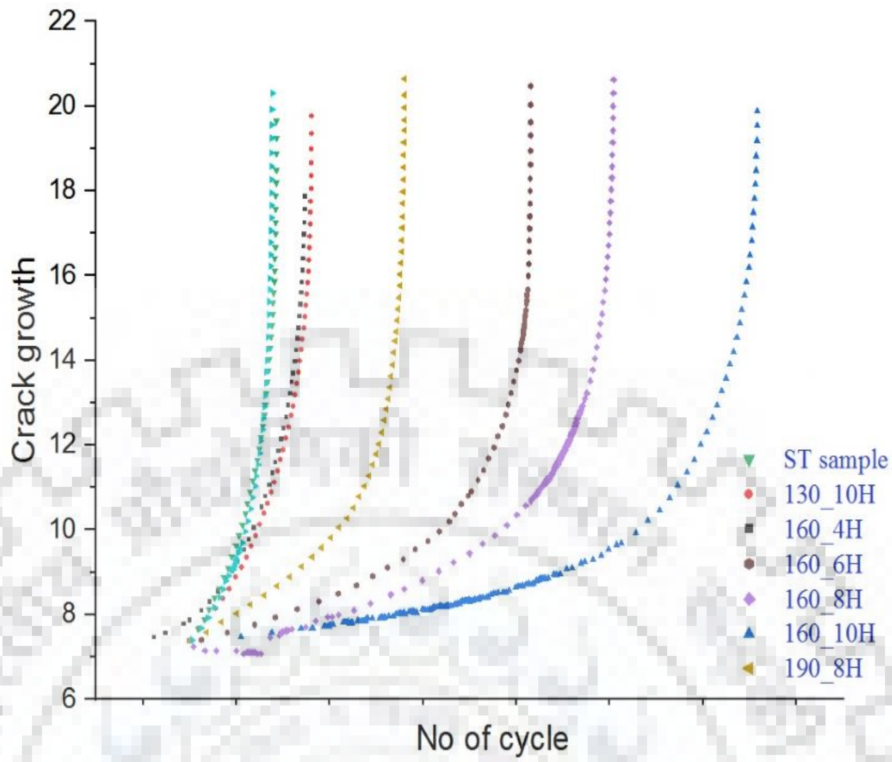


Fig 3.19 Crack growth against no of cycle plot obtained for solution treatment and different ageing parameter.

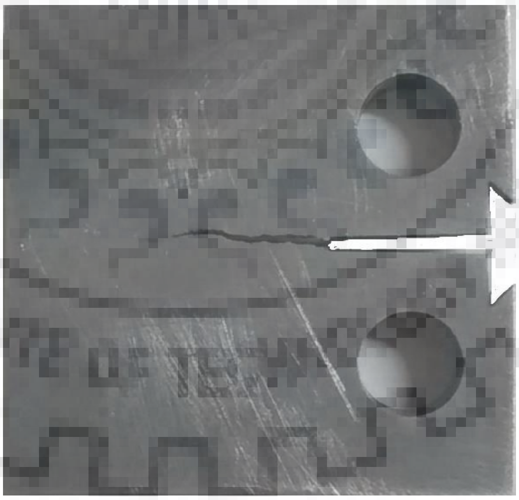


Fig 3.20 FCG sample after testing (representing the development of crack path.

Fig 3.19 represent the plot of crack growth obtained against no of cycle for different ageing parameters. It clearly reveals that there is a resistance hike along with the ageing parameter. However, the results are better at ageing at 160°C than comparison to ageing at 130°C and 190°C. As the diagram reveal.

Chapter 4
Results and Conclusions

- Ageing at temperature 130°C has no significant result on the mechanical properties of AA2014.
- Ageing at 190°C initially shows hike in strength up to 8Hour of ageing. The peak strength was found to be 475MPa. However, a drop observed for 10Hour. Ductility (in terms of %El) found to decrease with increasing time period of ageing. After 10Hour of ageing the ductility value observed to be 5.042%.
- The drop-in result obtained may be a result of over-ageing.
- Ageing at temperature 160°C get a peak strength of 490MPa. The results show greater strength than ageing at 190°C. A drop in ductility is observed throughout ageing at 160°C. however, the value of ductility remains better then ageing at 190°C temperature. Ductility value corresponding to the maximum strength is observed as 6.231%.
- FCG result at 130°C shows no significant difference with solution treatment sample. A resistance in crack growth observed for ageing at temp 160°C temp. the hike was continuous up to 8Hour of ageing but no further. A max resistance is obtained at 160_8H ageing parameter. Ageing at 190°C shows a slightly hike in resistance initially but no further.
- Better mechanical properties are observed at low temperature (160°C) ageing.
- The crack growth path in case of ST sample are is obtained nearly a straight line whereas in case of aged samples these paths are zig-zag or deviated.
- The deviation in path may be a result of resistance of nucleated precipitates particles due to ageing.

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