STUDY OF THE AGE HARDENING BEHAVIOR OF Al - 4.5Cu/$ZrSiO_4$ COMPOSITE IN DIFFERENT QUENCHING MEDIA

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ASHUTOSH
Roll No.6050501

Under
the able guidance of
Dr. Sanjeev Das
School of Physics and Material Science
Thapar University, Patiala

School of Physics & Material Sciences
Thapar University, Patiala
Patiala - 147001
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Dedicated

To

My Grand Parents

CERTIFICATE

This is to certify that Mr. ASHUTOSH, Roll No. 6050501 has worked on this thesis report as a final fulfillment for award of the degree of MASTERS IN TECHNOLGY in Material Science and Engineering. I certify that the matter embodied in this report is of the candidate's own record and not submitted to any other university in any part or full form for the award of such kind of a degree.

(Dr. Sanjeev Das)
Supervisor
SPMS, Thapar University
Patiala

Countersigned by:

Dr.O.P.Pandey	**Dr. R.K.Sharma**
(Prof. & Head)	**Dean of academic Affairs**
School of Physics and Materials Science	**Thapar University**
Thapar University, Patiala	**Patiala**

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Ashutosh
Roll No.6050501

ABSTRACT

This report deals with a comparative study based on the age hardening behavior of Al-4.5%Cu metal matrix composite reinforced with zircon sand particulates in different quenching media viz, water, oil, and salt brine solution (7%). All the samples were solutionised; quenched in water, oil, and salt solution, and aged in a vacuum oven. Vicker's microhardness test is conducted on all samples for ageing. Optical micrographs of the as cast alloy composite indicates that the matrix of the composite has the cellular structure. Segregation of copper rich phase ($CuAl_2$) has been found in the vicinity of particle matrix interface. Scanning electron micrographs at low magnification show uniformly distributed zircon particles throughout the matrix. Scanning electron micrographs at higher magnification shows the presence of uniformly distributed secondary phase in the alloy matrix indicating a strong continuous bond between zircon particle and the alloy matrix. Results of ageing kinetics demonstrate that the microhardness of age hardenable Al-Cu alloys depend on the rate on which the alloy is cooled after solution heat treatment. The peak hardness in oil quenched sample is attained at an early stage while water quenched sample show peak hardness at a later stage. Salt brine quenched sample shows the maximum hardness but peak hardness is in between water and oil quenched samples. Thermal cycling of the composite has been studied in different quenching media. A maximum of different heat treatment cycles are taken and microhardness of the matrix is discussed and an interesting hardness result were observed while varying quenching media.

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LIST OF ABBREVIATIONS

AAA	Aluminium Association of America
AMC	Aluminium Matrix Composites
BCT	Body Centered Tetragonal
EDS	Energy Dispersive X-ray Spectroscopy
CTE	Coefficient of Thermal Expansion
Hv	Vicker’s hardness
IADS	International Alloy Development System
GP Zones	Guinier Preston Zones
MMC	Metal Matrix Composites
PRMMC	Particulate Reinforced Metal Matrix Composites
SEM	Scanning Electron Microscope
SSSS	Super Saturated Solid Solution
UTS	Ultimate Tensile Strength
YS	Yield Strength

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Chapter 1

INTRODUCTION

Aluminium alloys are widely used in aerospace and automobile industries due to their low density and good mechanical properties, better corrosion resistance and wear, low thermal coefficient of expansion as compared to conventional metals and alloys. The excellent mechanical properties of these materials and relatively low production cost make them a very attractive candidate for a variety of applications both from scientific and technological Viewpoints. Recently, Aluminium metal matrix composites with Al_2O_3, SiC whisker and Ceramic fiber used to substitutes steel [1]. Some of these materials have been used to manufacture automotive components for trial but its commercialization was limited due to the difficulties in manufacturing and also the high production cost. On the other hand particulate reinforced composites have been reported to have a better plastic forming capability than whisker & fiber reinforced composites [2], and thus have an advantage to lower the manufacturing cost. Also they exhibit excellent heat and wear resistances of due to the superior hardness and heat resistance characteristics of the particle distributed in the matrix [3-5]. Moreover they are easily available and synthesized via a variety of manufacturing routes. There are several fabrication techniques are available for the in manufacturing of MMC materials. According to the type of reinforcement, the fabrication techniques can vary considerably. These techniques include stir casting [6-8], liquid metal infiltration [9], squeeze casting [10], and spray co deposition [11]. Stir casting route is generally practiced commercially [12-13]. Its advantage lies in its simplicity, flexibility and applicability to large quantity production. Numerous research work have been reported on Aluminium reinforced with various particulates such as SiC, B, TiC, Si_3N_4, Silica Sand, MgO, mica glass beads, B_4C, Al_2O_3 [5,14,15]. Limited work has been reported on fabrication and Characterization of zircon sand reinforcement in Aluminium matrix alloys. Zircon sand possesses high hardness, high modulus of elasticity, and excellent thermal stability. Banerji et al. found improvement in hardness, abrasive and wear resistance, elastic modulus, 0.2% proof stress, and UTS of cast Al-3wt%Mg alloy due to dispersions of zircon sand particles [16], which makes zircon sand reinforced composite an important material for industries like aerospace, military, automobiles etc. Also the thermal coefficient of expansion of zircon sand is comparable to that of SiC. Hence the influence of zircon sand reinforcement in ageing behavior of Al-4.5%Cu matrix is really interesting.

Cast Al-Si-Mg alloys have been widely used in automotive and aircraft industries for their good properties and high strength-to-weight ratio [17,18]. Intensive studies of this cast aluminum family have been found in the literature in terms of enhancing the mechanical properties [18, 20-24]. It is well known that the heat treatment is one of the important methods for improving the mechanical properties of aluminum alloys [21]. The heat treatment of agehardenable aluminum alloys involves solutionzing the alloys, quenching, and then either aging at room temperature (natural aging) or at an elevated temperature (artificial aging) [18].

Quenching is a crucial step to suppress the precipitation to retain the supersaturation of solid solution, control the distortion, and minimize the residual stress in aluminum alloys. Quenching media commonly used for aluminum alloys include brine solution, water, and polymer solutions [25-26]. The physical properties of polymer quench bath directly affect the cooling rate of a quenched part. These properties include the type of quenchant, its temperature, concentration, and agitation level [25-27]. These parameters must be controlled to optimize the quenching process in terms of alloy microstructure, properties, and performance. People have used polymer quenchants and oils to still maintain similar quenching performance [25-28].

The present investigation has been done to study the age hardening behavior of Al-4.5%Cu alloy reinforced with zircon sand particles, synthesized by stir casting route in different quenching media water, oil and salt brine solution (7%). Current researches indicate an accelerated ageing behavior with Aluminium reinforced with zircon sand when quenched in water [29-30]. However, limited work has been done when the hardening behavior of Al-Cu alloy composites are characterized in different quenching media other than water. In present case effect of age hardening effect in oil and salt brine solution has been examined along with thermal cycling of composites.

Chapter 2.

LITERATURE REVIEW

2.1 Aluminium and its alloys

The three main alloys of importance in engineering and aerospace applications are the aluminium alloys, steels and nickel alloys. Titanium, magnesium and copper alloys are also significant. Aluminium alloys with great durability and high strength, some with a tensile strength as good as that of constructional steels, are available to the designer in the form of extruded profiles, rolled sheet and plate, castings and forgings. The majority of these alloys consist of aluminium with carefully controlled additions of copper, magnesium, silicon, manganese, zinc and more recently lithium.

Aluminium alloys are the dominant materials for airframe structures. There are three main classes of aluminium alloys used in aerospace applications, though only the wrought heat-treated alloys have sufficient strength for structural components. [31]

2.1.1 □□Casting Alloys: Aluminium and its alloys are used in a variety of cast and wrought form and conditions of heat treatment. Forgings, sections, extrusions, sheets, plate, strip, foils and wire are some of the examples of wrought form while castings are available as sand, pressure and gravity die-castings. e.g. Al-Si and Al-Mg alloys

2.1.2 □□□Wrought Aluminium Alloys: to meet various requirements, aluminium is alloyed with copper, manganese, magnesium, zinc, nickel and silicon as major alloying elements. These alloying additions improve the properties of aluminium when added in desired percentages. The AAA (Aluminium Association of America) has classified the wrought aluminium alloys according to a four-digit system. The classification is adopted by the International Alloy Development System (IADS) and by most of the countries in the world. Table 1-3[70] gives the basis of designation of wrought and cast aluminum alloys in the four-digit system. The first digit identifies the alloy type the second digit shows the specific alloy modification. The last two digits indicate the specific aluminium alloy or the purity level in case of pure aluminium.

Table 1- Designation of Wrought Aluminium alloys

Alloy Designation	Detail
1XXX	99% pure Aluminium
2XXX	Cu containing alloy
3XXX	Mn containing alloy
4XXX	Si containing alloy
5XXX	Mg containing alloy
6XXX	Mg and Si containing alloy
7XXX	Zn containing alloy
8XXX	Other alloys
9XXX	Unassigned

Table 2- Designation of Cast Aluminium alloys

Alloy Designation	Detail
1XX.X	99% pure Aluminium
2XX.X	Cu containing alloy
3XX.X	Si, Cu/Mg containing alloy
4XX.X	Si containing alloy
5XX.X	Mg containing alloy
7XX.X	Zn containing alloy
8XX.X	Sn containing alloy
9XX.X	Other alloys
6XX.X	Unassigned

The condition of temper of Aluminium alloys be denoted by specific letters as shown below

Table 3- Temper Designation System

Letter	Condition of alloy
F	As–fabricated
O	Annealed
H	Strain hardened (Wrought products only)
W	Solution heat treated
T	Thermally treated to produce tempers other than F, O, H
T4	Solution treated
T6	Solution treated and aged

Numeric additions indicate specific variations

2.1.3 Heat Treatable Aluminium Alloys: Heat treating in its broadest sense, refers to any of the heating and cooling operations are performed for the purpose of changing the mechanical properties, the metallurgical structure, or the residual stress state of a metal product.

When the term is applied to aluminum alloys, however, its use frequently is restricted to the specific operations employed to increase strength and hardness of the precipitation-hardenable wrought and cast alloys. These usually are referred to as the "heat-treatable" alloys to distinguish them from those alloys in which no significant strengthening can be achieved by heating and cooling [31-32].

The Aluminium alloys of this class belong to systems with limited solubility in solid state. These are precipitation hardenable alloys. The main characteristic of these alloys system is temperature dependent equilibrium solid solubility, which increases with rise in temperature. In addition the other requirements are possibility of retaining single phase supersaturated solid solution by quenching, and precipitation of coherent/partially coherent phase by decomposition of the super saturated solid solution. The examples of this group are:

1. Aluminum-copper systems with strengthening from $CuAl_2$
2. Aluminum-copper-magnesium systems (magnesium intensifies precipitation)
3. Aluminum-magnesium-silicon systems with strengthening from Mg_2Si
4. Aluminum-zinc-magnesium systems with strengthening from $MgZn_2$

2.1.4 Non-Heat treatable Aluminium alloys: These alloys do not respond to heat treatment, because they consist of a homogeneous solid solution with or without non-coherent precipitate(s) and show low strength and high ductility. These alloys may be stress hardened. Commercially pure Aluminium (1100), Al-Mn (3003), Al-Mn-Mg, and Al-Si alloys are examples of this class [31]. These alloys are used as sheet, bar, plates, wire, extrusion and so on. They are readily bent, formed and welded and possess excellent resistance to corrosion.

2.2 Age-hardening of aluminium alloys.

Hardening and strengthening of a metal alloy by extremely small and uniformly distributed dispersed particles that precipitates from a supersaturated solid solution. The alloy becomes harder with time or as it ages it develops hardness and strength. The hardness of the quenched alloy increases as a function of ageing time. Ageing of the quenched alloy at room temperature is known as natural ageing while at elevated temperatures is known as artificial ageing. Precipitation in solid solution occurs when the solubility of solute decreases with decreasing temperature. The precipitate of the second phase should be coherent in nature. The object of age hardening is to create in a heat treated alloy a dense of fine dispersions of precipitated particles in a matrix of deformable metal. The precipitate particles act as obstacles to dislocation motion and thereby strengthen the heat-treated alloy.

The strengthening mechanisms in wrought, heat-treated Aluminium alloys can be demonstrated using the Al-Cu alloy as shown in Fig.1

Precipitation hardenable aluminum alloys are heat treated in three-step process: solution heat treatment, quenching and age hardening. The solution treatment aims to dissolve the soluble phases. Quenching aims to preserve the solid solution formed by rapidly cooling to some lower temperature, usually room temperature. Age hardening aims to precipitate out the strengthening phases. Overheating above or under heating below a specfied range of temperatures in the solution treatment and age hardening steps as well as inadequate quenching may cause degradation of mechanical properties [31].

Hardening is achieved by the controlled rejection of copper from a supersaturated solid solution. The solubility of copper in α-Aluminium increases with increasing temperature up to the eutectic temperature of about 540°C. The equilibrium microstructure below the eutectic temperature is a two-phase mixture of α-Aluminium and the Al_2Cu intermetallic phase. Rapid quenching from the solution temperature prevents the kinetically slow precipitation of θ, forming a highly supersaturated solid solution of copper.

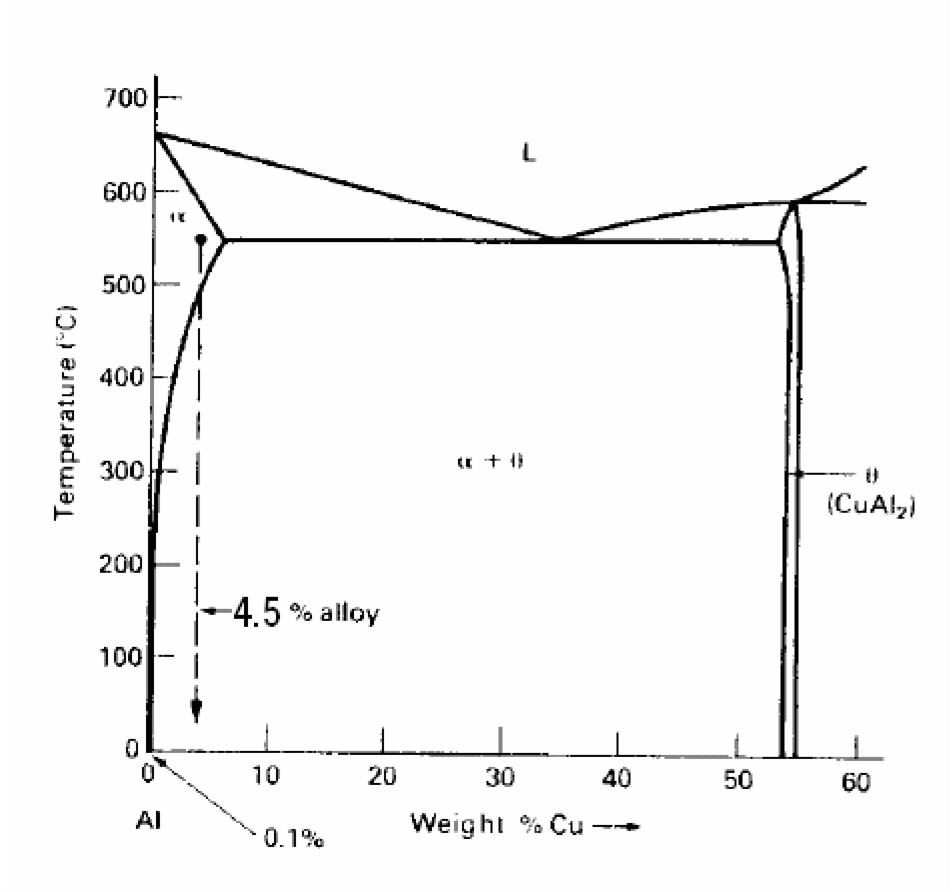


Fig.1 - The binary phase diagram for Aluminium-Copper alloy [33].

2.3 The Effect of Ageing on Microstructure and Strength.

Ageing may occur at room temperature (Natural Ageing) or above (Artificial Ageing), and takes place via a sequence of precipitation reactions. The precipitation sequence is driven by the super saturation of copper in solution.

During ageing, copper atoms segregate by diffusion to form copper-rich zones called Guinier-Preston GP(1) zones. The difference in atomic size of copper and aluminium strains the lattice. Hardening is due to the increased work required to move dislocations through the strained lattice (coherency stress) and work required for dislocations to cut though the GP(1) zones (cutting stress). Above around 100°C, further copper segregation produces GP(2) zones, sometimes known as θ" phase.

Hardening occurs by the same mechanisms as the GP(1) zones, though GP(2) zones have a stronger effect. A high number density of GP(2) zones or θ" gives the maximum strengthening effect.

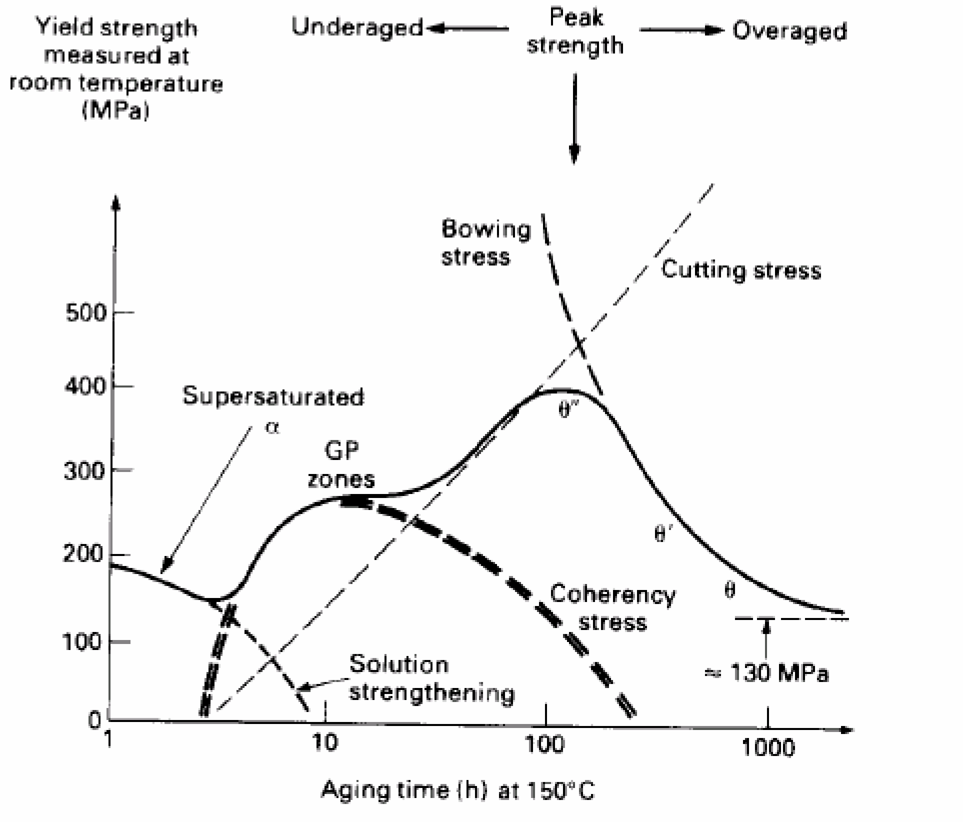


Fig. 2 - The effect of ageing time on the yield stress of an aluminum-copper alloy, aged at 180°C, showing the age hardening strengthening effects [34].

Further growth of the GP(2) zones leads to the formation of the θ' phase. θ" is the dominant phase at the peak hardness, where strengthening is due to a combination of lattice coherency strain (coherency stress), precipitate cutting by dislocations (cutting stress) and dislocation bowing between precipitates (bowing stress). With further ageing, the precipitate distribution changes as large, widely dispersed precipitates grow at the expense of finely dispersed small precipitates. This process is known as "**Ostwald**

ripening". Dislocation bowing between precipitates becomes easy and the strengthening contribution from coherency strain and precipitate cutting is lost. Over-ageing develops the equilibrium tetragonal θ phase (Al_2Cu), which does not have a strong strengthening effect.

2.4 Applications of aluminium alloys: aluminum is a strong, durable and lightweight metal. In today's energy-conscious society, these three basic properties combine to make the metal the preferred material of construction for transport applications, where weight reduction to reduce fuel consumption and to increase load carrying capacity is vital. Products like motorcars, aircraft, ships, conductors, lorries and trains are all obvious examples[35].

Electrical Conductors

Conductors in either the 1000 or 6000 series alloys are sensible technical alternatives to copper for all electrical conductors, even in domestic wiring.

Transport

Aluminium and its alloys have been the prime material of construction for the aircraft industry throughout most of its history. Even today, when titanium and composites are growing in use, 70% of commercial civil aircraft airframes are made from aluminium alloys, and without aluminium civil aviation would not be economically viable.

Packaging

The successful use of the 1000 series alloys as foil for food wrapping and for containers utilizes their good corrosion resistance and barrier properties against UV light, moisture and odour. Foil can be readily formed, attractively decorated and can be usefully combined with paper and plastic if required.

Building and Architecture

Aluminium is used in buildings for a wide spectrum of applications. These include roofing for factories which incorporate foil vapour barriers, windows and pre formed sheet cladding features, doors, canopies and fronts for shops and prestigious buildings, architectural hardware and fittings, rainwater goods and replacement windows.

In composites, Particle reinforced AMCs are in use as recreational products including golf club shaft and head, skating shoe, baseball shafts, horseshoes and bicycle frames. AMCs containing high volume fraction ceramic particles are being used as microprocessor lids and integrated heat sinks in electronic packaging. They are also in use as carrier plates and microwave housing.

2.5 Metal Matrix Composites: An alternative to conventional alloys are metal matrix composites (MMC's), which have a high specific modulus, good wear resistance and a tailorable coefficient of thermal expansion. A major drawback to these MMC's is high cost.

A metal matrix composite (MMC) combines into a single material a metallic base with a reinforcing constituent, which is usually non-metallic and is commonly a ceramic. By definition, MMC's are produced by means of processes other than conventional metal alloying. Like their polymer matrix counterparts, these composites are often produced by combining two pre-existing constituents (e.g. a metal and a ceramic fibre). Processes commonly used include powder metallurgy, diffusion bonding, liquid phase sintering, and squeeze-infiltration and stir casting. [9-11]

Major Phases: The major phases in a metal matrix composite are one or several reinforcements (strengthening phases) and a matrix, which transfers external load to the reinforcement(s). In AMCs, these can be briefly described as follows.

2.5.1 Reinforcement

The most common reinforcements are silicon carbide (SiC), alumina (Al_2O_3), and graphite. Other carbides such as TiC and B_4C as well as aluminum nitride (AlN) can be used. SiC is of interest because of its relatively high modulus, low density, and its availability in different reinforcement shapes and sizes [36]. Three different reinforcement shapes are considered: particulates, whiskers or discontinuous fibers, and continuous fibers. AMCs with particulates offer low cost, isotropic properties, and significant stiffness improvement, but the strength, strain to failure, and fracture toughness are low com-pared to the matrix material. AMCs with discontinuous polycrystalline fibers or flakes offer higher strength. Monocrystalline whiskers have much higher strength, but their cost is higher. Composites with continuous fibers offer the best combination of strength and stiffness, but their cost is relatively very high. Fibers such as boron (as Borsic), carbon (as Pitch), alumina (as Safill and Safi- max), and SiC are considered for strengthening AMCs[36-40].

2.5.2 Matrix

According to the aluminum association system, the matrix materials generally employed include pure Al (AA 1000), AlCu (AA 2000), AlMg (AA 5000), AlMgSi (AA 6000), AlZn (AA 7000), and AlLi (AA 8000) [41]. In particular, aluminum alloys in the Al–Si and Al–Cu systems are successful matrices for AMCs. Heat-treatable Al alloys are interesting because with post processing, additional improvement in mechanical properties can be obtained. .

2.6 Al-MMC's: Aluminium alloys are widely used in aerospace and automobile industries due to their low density and good mechanical properties, better corrosion resistance and wear, low thermal coefficient of expansion as compared to conventional metals and alloys. Of all the AMCs, particle reinforced AMCs constitutes largest quantity of composites produced and utilized on volume and weight basis. PAMCs are produced by PM stir cast/melt infiltration/spraying/in situ processing techniques at industrial level. Particulates of SiC, Al_2O_3, TiC, TiB_2, B_4C have been used as reinforcements [5,15,42]. In general, PRMMCs are at least approximately isotropic. They are produced using both solid state (powder metallurgy)[43] and liquid metal techniques (stir casting etc,)[30]. Their mechanical properties, while often inferior to those of fibre-reinforced metals, are more or less isotropic and often represent, at moderate cost, significant improvements over those of corresponding unreinforced metals. Their novelties, and their interesting mechanical behavior (at both micro- and macroscopic levels), have led to many publications, exploring many features of their microstructure, deformation, and fracture behavior. Many mechanisms responsible for their mechanical characteristics are now well understood, including the roles of damage development, internal stresses, reinforcement clustering, interfacial bond strength and the effects of the presence of the reinforcements on ageing of the matrix.

A major difficulty when studying the literature data on Aluminium based metal matrix composites is the wide range of information that is available. This is mainly due to the facts above that the materials are a combination of two or more components. This chapter presents a review of the literature data available on the effect of various reinforcement types, their size and volume fraction, ageing behavior with Al based MMC's. Metal matrix composites are a combination of two phases, matrix and the reinforcement. Matrices can be selected from a number of Aluminium alloys e.g. 2000, 6000, 7000, and many reinforcement types SiC, B_4C, Al_2O_3, AlN, and C etc. are available in different sizes, morphologies (particulates, short fibers, long fibers and platelets) and volume fractions. These reinforcements can be combined with the different matrices, resulting in large composite systems. Furthermore, several different processing routes, such as powder metallurgy, square casting, rheocasting etc, can also process a specific MMC. An additional factor for a number of MMC's is also that their mechanical properties depend upon the age hardening behavior of the matrix alloy.

2.7 Age Hardening in Al-MMC's

Age hardening in Al-MMC has been of great interest of present research. The nature of change in kinetics and magnitude of hardening has been investigated by the authors for a various type of reinforcements in Al-MMC. Authors have shown that the ageing characteristic accelerates with the reinforcement as compared to the unreinforced alloy.

The effects of artificial ageing on wear properties of AA6063 Aluminium alloy reported that ageing treatments improved the wear behavior of the matrix alloy as compared to the as cast samples. The artificial ageing produces the harder structure that is attributable to acceleration of precipitation of Mg_2Si and other phases as $CuAl_2$ and AlFeSi[44].

In the case of Al-Mg-Si Alloy (6xxx series) based composites it has been shown that the presence of ceramic reinforcements such as SiC/Al_2O_3 (whisker, short fibers or particles) lead to acceleration in ageing kinetics when compared to unreinforced alloy [45,46].

However in contrast different rules have been reported in the case of hardening behavior of ceramic reinforced Al-Cu alloy composites. Suresh et al [47] reported rapid age hardening of Al-3.5Cu composites during the ageing at 190°C as compared to unreinforced alloy. On the contrary, Kim et al [48] reported significant retardation in the kinetics of hardening (i.e. increased time to peak hardness) during ageing of Al-4 Cu-SiC whisker composites (1992).

However, Harris et al [49] have shown that SiCp additions have no influence on room temperature ageing kinetics of Al-4Cu-SiCp Composites as compared to the unreinforced alloy. Further more they also reported that, in case of high temperature ageing (ageing at 135°C, 170°C & 190°C), the composites attain peak hardness much earlier as compared to unreinforced alloy. Dutta et al [50] observed the same behavior on the cast and extruded Al-3.2Cu-5vol%SiCp composites exhibited accelerated hardening during the natural and high temperature ageing. While there have been many papers examining the abrasive wear properties of MMC's only some authors have studied the effect of thermal ageing on wear resistance. W.Qsong et at [51] examined the behavior of thermal ageing on the abrasive wear resistance of 2014 Al-SiC and 6061Al-SiC composites. They found that raising the ageing temperature from the underaged to peakaged condition improved the wear resistance of the composite with decreasing the size of particulates.

M.Gupta et al [42], examined an Al-Based MMC Al-2%Cu reinforced with graphite and SiC using conventional casting techniques. They demonstrated accelerated ageing kinetics in samples of Al-Cu/SiC when compared to Al-Cu/C samples, this is attributed to the enhanced Cu-enrichment in the interfacial region when compared to C-reinforced samples, which favors the hardening behavior. Similar behavior is observed by M.Gupta and M.K Sarappa [52] in 1995,that if the varying SiC volume fraction is reinforced in an Al-based alloy (6061/SiC) ageing kinetics increase with increasing volume fraction of SiC particles and decreasing their sizes. L.Salvo et al [53], studied the various types of reinforcements including SiC of different sizes and volume fraction (up to 50%), chopped nicalon and safill in 6061 and reported the same lines.

Vijay K. Sharma et al [54] has also reported the behavior of Al-Cu-Mg alloy composite with SiC reinforcements with varying sizes and got the similar results. N V Ravi Kumar et al [55] chosen the alloy Al-Zn Mg/SiCp and examined the matrix strength on mechanical properties of composite and showed that the strengthening was found to be dependent on the damage that is occurring during strain (with different heat treatments). Similar behavior is observed by stir cast 6061/SiCp composite [52]. Compared to control alloy the UTS values had decreased in peak and overage condition whereas in solutionised condition different trends were observed in compositions having higher yield strength than in tension. The fracture was found to be a combination of particle fracture and particle pull out.

An accelerated ageing response has also been reported with SiC particulate whisker reinforcements in SiCp/8090[56,45], SiCw/Al-Li[46], and SiCw/Al-Li-Cu[57], SiCw/Al-Li-Cu-Mg-Zr composites [58]. Authors have also reinforced Al-4%Cu with Al_2O_3 fibers produced by squeeze casting. The Cu precipitation in al-4Cu alloy is different from that of the unreinforced alloy [43]. The formation of GP zones have been find to be inhibited by the presence of fibers while the precipitation of θ' phase is accelerated in composites. The elastic moduli of the composites have been confirmed to be higher than that of the unreinforced alloy, and sensitive to age hardening. S.Arakawa et al [43] investigated the effect of heterogeneous precipitation of Al-4Cu/Al_2O_3 produced by mechanical alloying. The authors have found that the age hardening response is significantly decreases, and that considerable θ-phases are formed at a very short ageing time, as compared to those by powder or ingot method. This is attributed to the preferential precipitation of the stable θ-phases on grain boundaries occurs in earlier stages of ageing, and extends to fair amount due to very fine grains, which reduces the concentration of Cu atoms in matrix to form GP Zones and intermediates. There is a remarkable ageing kinetics in mechanically alloyed composite, which is ascribed to the precipitation of intermediates assisted by the higher dislocation density.

Mandal et al [59] reported the effect of TiB_2 particles in Al-4Cu alloy. They also showed a significant enhancement in ageing kinetics of the alloy in presence of TiB_2 particles in both as cast and peak aged conditions. However with higher additions of TiB_2 particles' strengthening is marginally reduced and the formation of Al_3Ti phase is completely suppressed. In contrast, strengthening contribution from TiB_2 particles increases in amount of these particles as expected.

In all such type of investigations, the authors have selected the various ceramic reinforcements, in the current research has been constricted to few reinforcement types such ac Al_2O_3, SiC, B_4C. Limited work has been reported on fabrication and characterization of Aluminium zircon composite. Banerji et al observed that the abrasive wear rate of the Al-Si-Mg alloy decreases with in volume fraction (>0.09) of zircon particles [16].

Current researches shows that the ageing behavior of Al-4.5%Cu matrix alloy reinforced with zircon sand particles of varying sizes accelerated ageing response as compared to the matrix alloy [29,30]. However it was found that Al_2O_3 reinforced composites show higher acceleration in age hardening as compared to zircon sand particles, which is due to the breakage of Al_2O_3 particles during melting and stirring processes. We se in all the research here on Aluminium metal matrix composites the age hardening is better age compared to the monolithic alloy due the enhanced dislocation density of the matrix as a result of the difference of CTE between matrix and the reinforcement. Dislocations generated due to the CTE mismatch act as heterogeneous nucleation sites for the precipitates. If we go through the literature we see little research on zircon sand though it possess high hardness, high modulus of elasticity and excellent thermal stability. Banerji et al [16] found improvement in hardness, abrasive wear resistance, elastic modulus, 0.2% Proof stress and UTS of cast Al-3wt%Mg alloy due to dispersions of zircon sand particles, which make zircon sand reinforced composite an important material for industries like aerospace, military, automobiles etc. Also, the coefficient of thermal expansion of zircon sand is comparable to that of SiC. Hence the influence of zircon sand in ageing behavior of Al-4.5%Cumatrix alloy composite seems to be interesting. Also limited work has been done to investigate ageing behavior of Al- alloys in oil and salt brine quenching media.[25-28]

2.8 Basic Requirements for Age Hardening: Two basic requirements are:

- The main basic requirement of age hardening alloy system is that the solid solubility limit should decrease with decrease in temperature, i.e. the phase diagram should show a solvus as illustrated in Fig 3
- The precipitate of the of the second phase should be coherent in nature.

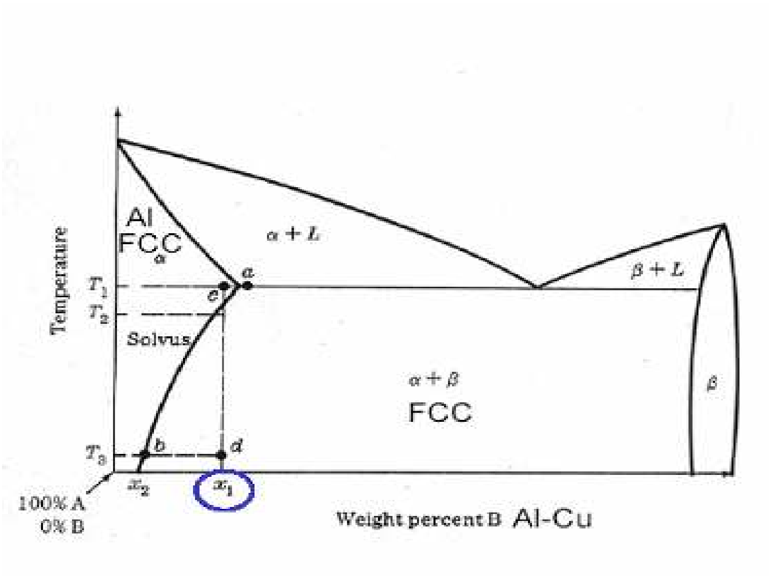


Fig. 3 - Hypothetical phase diagram for a precipitation hardenable alloy of composition x_1

Consider the precipitation hardening of an alloy of the composition x1 (since there is a large decrease in solid solubility of solid solution □ in decreasing temperature) from T2 to T3, the hardening involves three basic steps: ---

1. Solution heat treatment: the alloy sample is heated to a temperature between solvus and solidus temperature and soaked there until a uniform solid solution structure is produced.

2. Quenching: the sample is rapidly cooled to a lower temperature, usually room temperatureT3 to form supersaturated solid solution of □ phase. As shown in Fig 3 and 4 respectively.

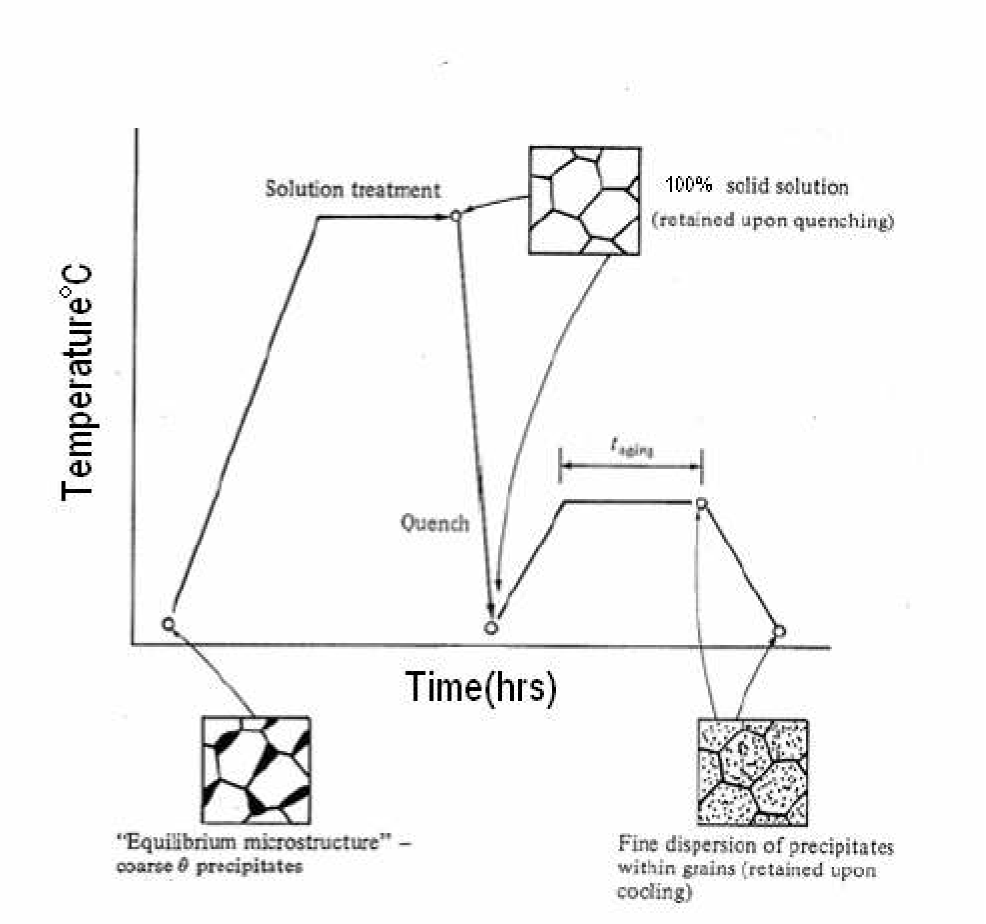


Fig. 4 - Schematic temperature versus time plot showing both solution & precipitation heat treatments for age hardening [67]

3. Ageing: ageing the solution heat-treated and quenched alloy sample is necessary so as to form a finely dispersed precipitate. These fine precipitates in alloy impede dislocation movement during deformation by forcing the dislocations to either cut through the precipitates or go around them. By restricting the dislocation motion during deformation, the alloy is strengthened. The ageing curve is shown as in Fig. 5

2.9 Ageing Curve: The microstrutural evolution and age hardening is given in the form of ageing curves. Ageing curves usually represent the effect of ageing time on strength and hardness of a precipitation strengthened that has been solution treated and quenched. The reduction in strength that occurs after long periods of time is known as overaging as shown in Fig: 5.

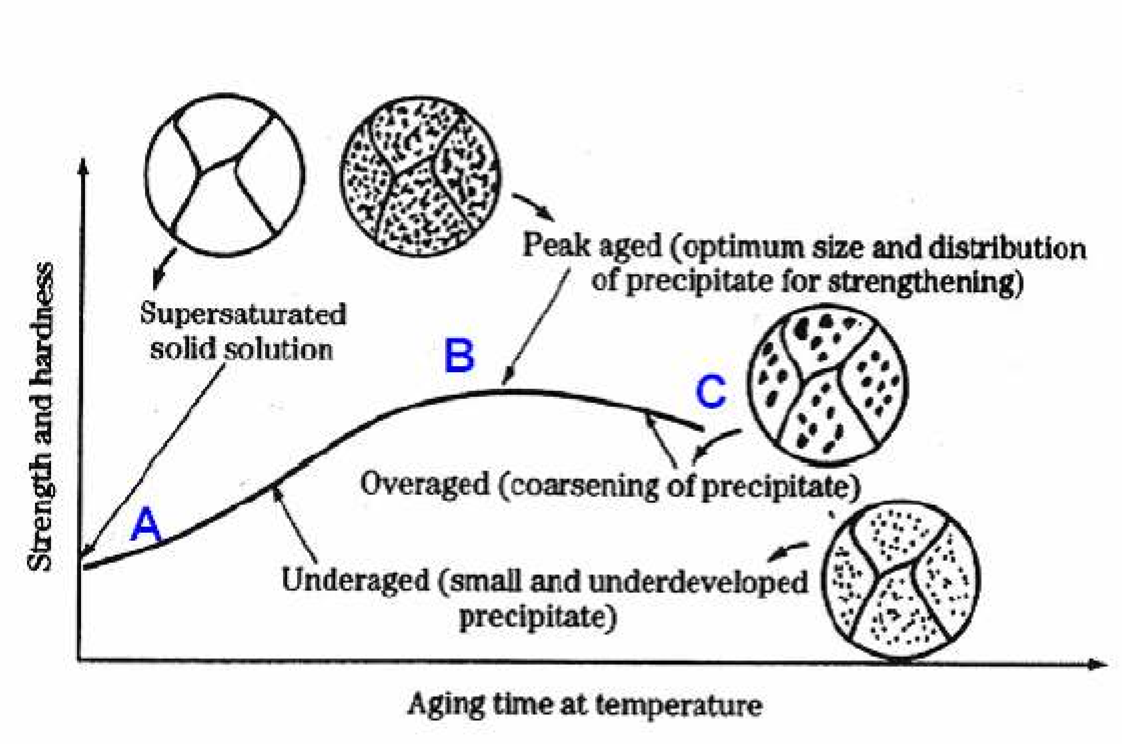
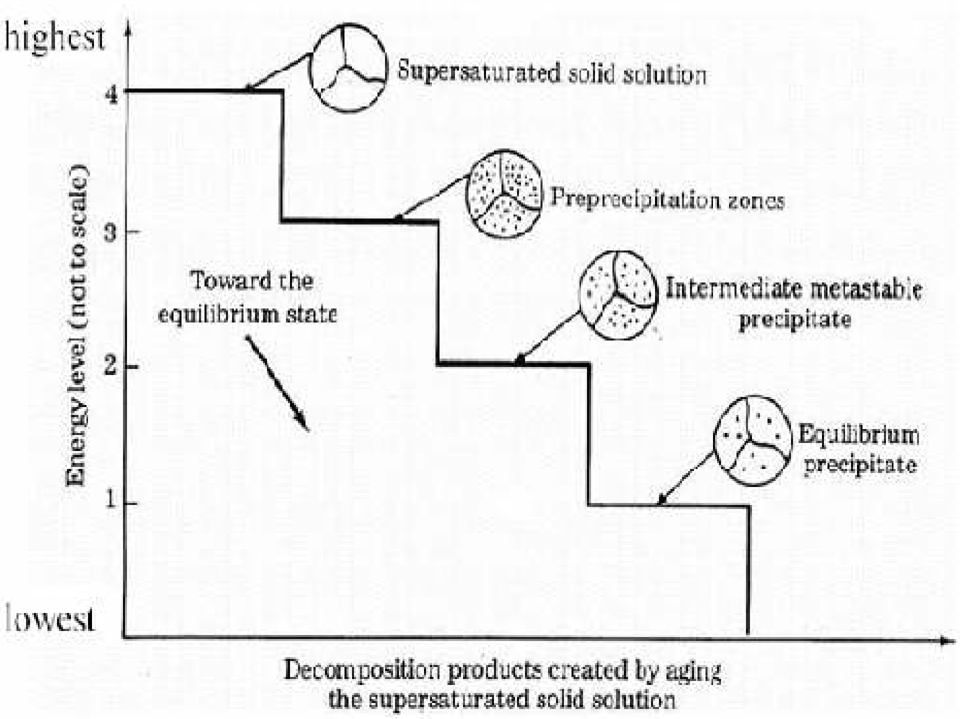


Fig. 5 - Schematic ageing curve for an Al-Cu alloy composite[33]

2.9.1 Effect of aging time and temperature: Decomposition products are created by the ageing of a supersaturated solid solution of a precipitation hardenable alloy, the highest energy is for the supersaturated solid solution, and the lowest energy level is for the equilibrium precipitate. The alloy can go spontaneously from a higher energy level to a lower one if there is sufficient energy of activation for the transformation and if the kinetic conditions are favourable. The supersaturated solid solution state is highly unstable and the alloy tends to seek a lower energy state by the spontaneous decomposition of the supersaturated solid solution into the metastable phases or the equilibrium phase.



SSS-α → GP1 Zone → GP2 Zone (θ'' phase)

↓

θ'- phase

↓

θ-phase (CuAl2)

Fig.6 (a) Decomposition Products created by ageing the supersaturated solution[33], (b) Precipitation sequence in Al-Cu alloy

GP1 Zone: these precipitate zones are formed at lower ageing temperatures and are formed by copper atoms segregating at SSS-□. These segregated regions have a shape of disk of few atoms thick and 8-10nm in diameter. GP1 Zones are said to be coherent with the matrix lattice as shown in Fig: 7 since Cu atoms just replace Al atoms in the lattice.

GP2 Zones: these precipitation zones have a tetragonal structure and are coherent with much larger in size than GP1 Zone formed in due course with time.

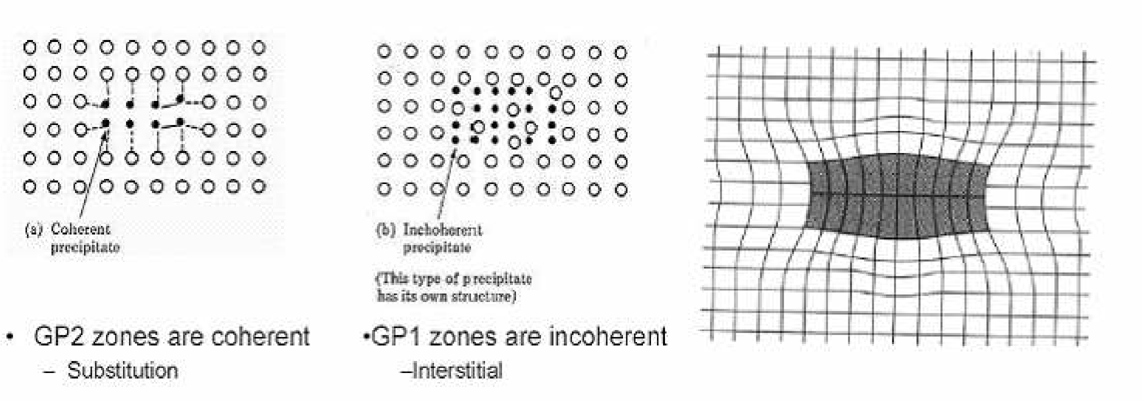
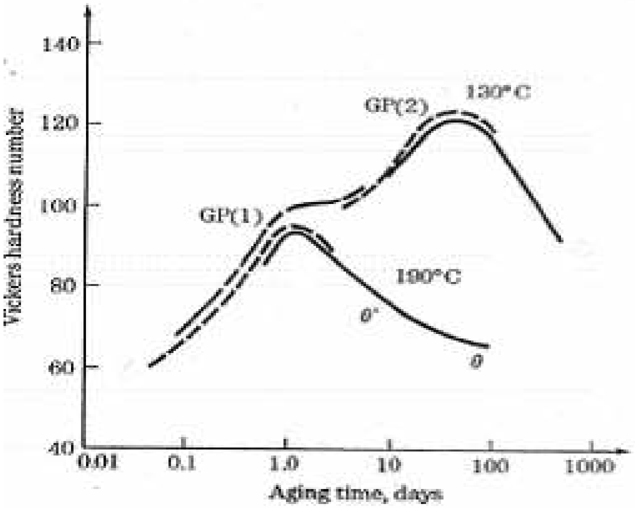


Fig.7(a) Coherency of the precipitates, (b) Geometry of Guinier Preston Zones[33]

θ'-phase: this phase nucleates heterogeneously on dislocations and are incoherent with the matrix as shown in Fig: 7, have a tetragonal structure with a thickness of 10-150nm.

θ-phase: this is the equilibrium phase incoherent and composition $CuAl_2$.this has the BCT structure and form from the θ' or directly from the matrix.

2.9.2 Hardening of GP zones



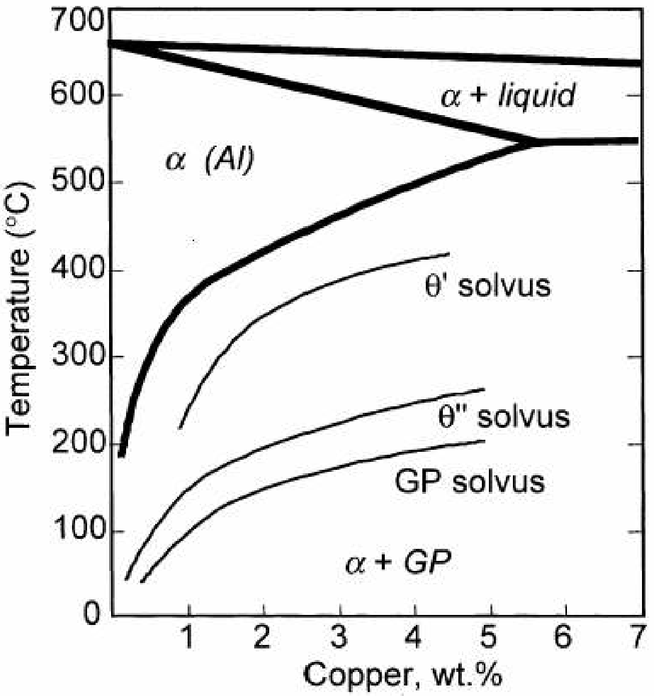
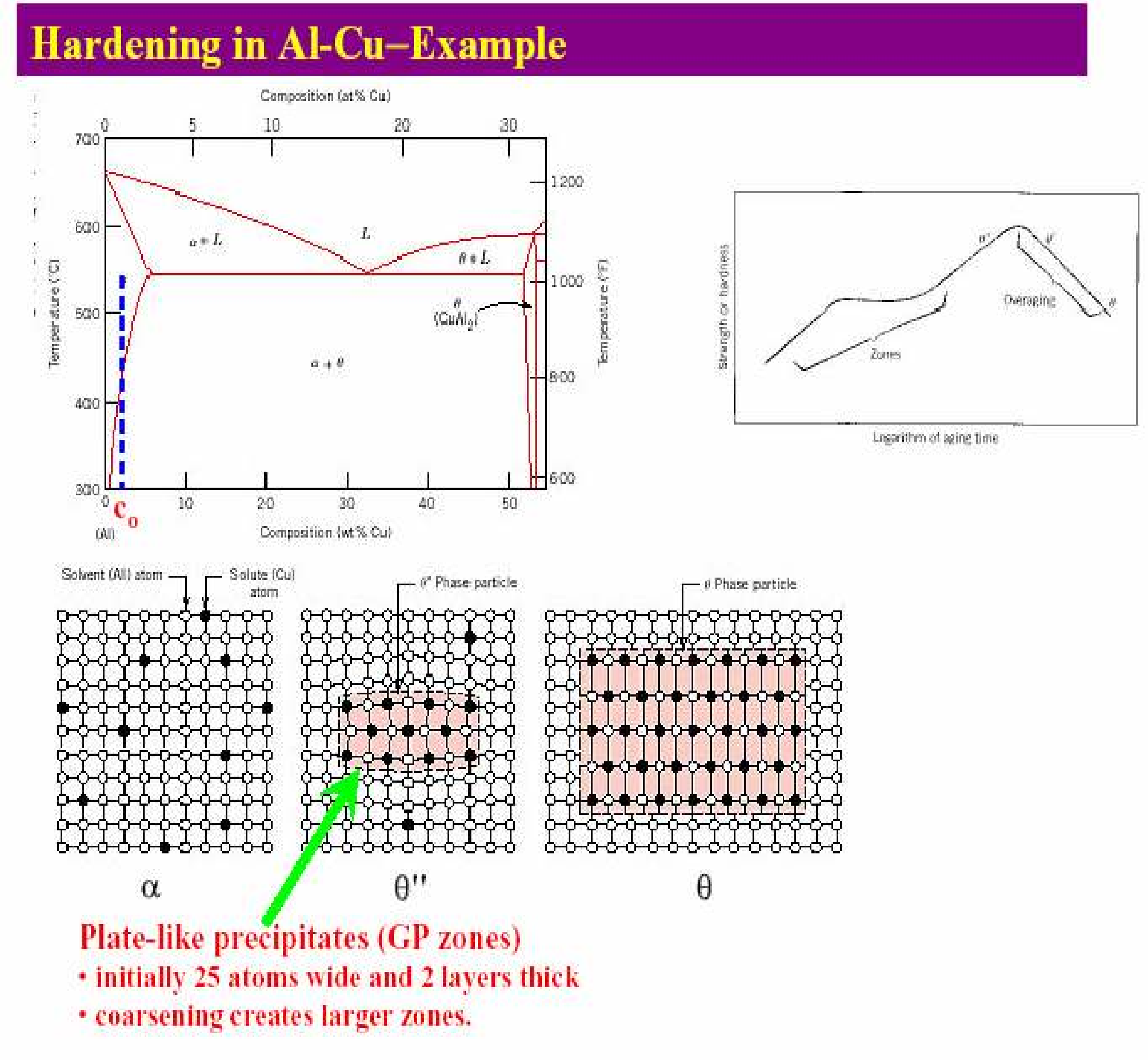


Fig. 8(a) Hardening of different zones [71], (b) Al-Cu phase diagram along with metastable phase boundaries for GP zones, θ' and θ", together with the equilibrium solvus line for the θ phase.[33,68]

Figure 8(b) shows the Al-rich corner of the equilibrium Al–Cu phase diagram, and includes the metastable solvus boundaries for GP zones, θ "and θ'. Over many studies [4–29], it has been proposed that the decomposition sequence in this system contains one or more of the following processes:

Supersaturated solid solution: GP Zones → θ" → θ' → θ

The complete precipitation sequence can only occur when the alloy is aged at temperatures below the GP zone solvus Fig. 8(b). Various steps in this process may be suppressed by aging at temperatures close to or above the intermediate solvus temperatures. The first stage of hardening at 180°C is contributed to GP1 zones. After reaching a critical diameter of between 5 and 10nm, an incubation period commences, during which the zone size and the hardness remain constant [32]. Further aging results in a second rise in hardness, attributed to θ" precipitation. The formation of θ" is also followed by a shorter incubation period and the subsequent formation of the metastable θ' phase. Prolonged aging results in the formation of the equilibrium θ phase. Example of hardening mechanism is illustrated in Fig8(c) given below:



Hardening in Al-Cu–Example

Plate-like precipitates (GP zones)

- initially 25 atoms wide and 2 layers thick
- coarsening creates larger zones.

Fig. 8(c) - Example of hardening behavior in Al-Cu alloy [69]

2.9.3 Effect of quenching medium

The objectives of quenching are to suppress the precipitation during quenching and to retain solute atoms and quenched-in vacancies in solution . The best combination of strength and ductility is achieved from a rapid quenching. Cooling rates should be selected to obtain the desired microstructures and to reduce the duration time over certain critical temperature range during quenching, in the regions where diffusion of smaller atoms can lead to precipitation at potential defects [60].

The quenchants used for quenching aluminum alloys include water, brine solution and polymer solution [61-62]. Water used to be the dominant quenchant for aluminum alloys, but water quenching most often causes the distortion, cracking, and residual stress problems [63,64].

Traditionally there are two ways to tackle these problems; one method is to increase the water temperature so that the temperature gradient between water and the part being quenched can be reduced [63]. It is reported that the water temperature affects properties of cast aluminum alloy A356 subjected to T6 heat treatment once the water temperature exceeds 60-70°C, with UTS and YS being significantly more sensitive than ductility [60]. However, the distortion problem can't be effectively solved by this method. The other method is to use the polymer solution. Quenching in polymer solution is used more widely nowadays since varying the polymer concentration can effectively reduce the distortion problem and more uniform quench can be readily obtained [64, 65]. Although a high quench rate is essential to achieve the high strength, in many cases, such a quench rate can't be used due to problems of high internal stress and distortion. This is especially true for cast components with the complex shapes and thin sections. To ensure that the minimum required strength is obtained throughout a cast component, the effects of quench rate on the strength of casting alloys need to be understood.

For age-hardenable aluminum alloys, the goal of quenching is to suppress the precipitation of a secondary phase during quenching process without distortion and excessive residual stress. Quenching media commonly used for aluminum alloys include brine solution, water, and polymer solutions [60]. Cold water had been the dominant quenchant for heat-treating aluminum alloys. However, in many cases, cold-water quench produces unacceptable distortion or high residual stress due to high thermal gradients generated upon cooling .

These parameters must be controlled to optimize the quenching process in terms of alloy microstructure, properties, and performance. Some investigations have been carried out by the researchers to study the effect of water temperature and the concentration of polymer solution on the mechanical properties of wrought aluminum alloys. Emadi et al [65] reported that increasing the water temperature or using air quench reduced the cooling rate and increased the chance of precipitation of a secondary phase during quenching.

Oil baths are not generally used in the wrought Al-alloys yet a variety of applications available in heat treatment of steels [66]. However, such data is scarce for cast aluminum alloys in the literature and quantitative measurement of the effects from each individual process parameter is not available.

Chapter 3

EXPERIMENTAL DETAILS

3.1 Materials

Nominal composition of the alloy used in this study is 4.5wt%Al (bal.) reinforced with zircon sand particles with an average size of 65 µm, produced by stir casting route.

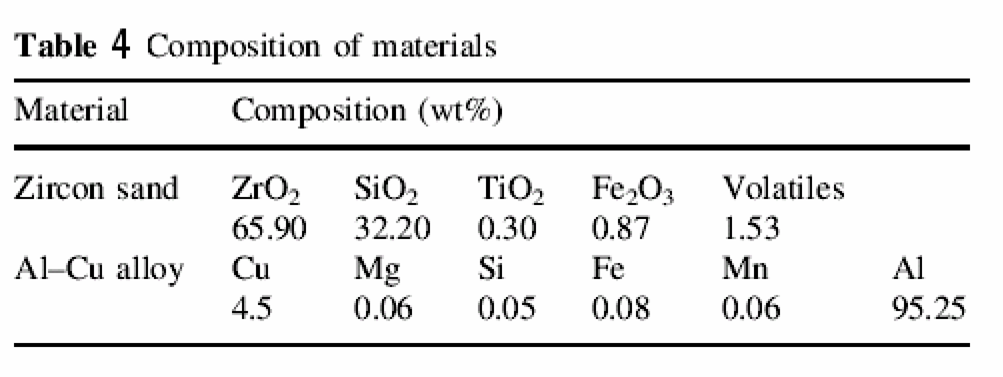


Table 4 Composition of materials

Material	Composition (wt%)					
Zircon sand	ZrO_2	SiO_2	TiO_2	Fe_2O_3	Volatiles	
	65.90	32.20	0.30	0.87	1.53	
Al–Cu alloy	Cu	Mg	Si	Fe	Mn	Al
	4.5	0.06	0.05	0.08	0.06	95.25

Quenching Media

Water, Transformer Oil, Salt brine solution (7%)

Experimental Techniques

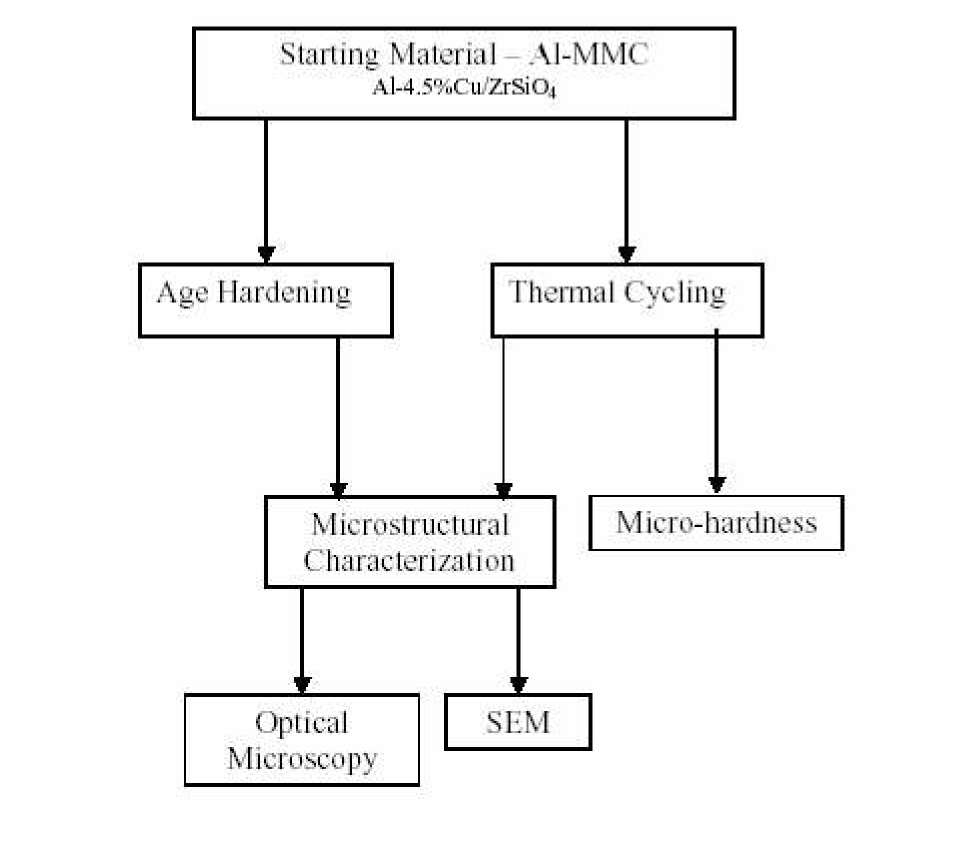


Fig. 9 - Experimental Techniques followed

3.2 X-Ray Diffraction and Microscopy

X-ray diffraction patterns of zircon particles and composite were recorded. Microstructural characterization studies were primarily accomplished using an optical microscope and scanning electron microscope (SEM) equipped with an energy dispersive X-ray spectrometer (EDS). The monolithic alloy and composite samples were metallographically polished and etched with Keller's reagent.

3.3 Ageing studies

The hardness measurements were done by Vickers microhardness tester. Five sets of readings taken for each sample for better accuracy.

The following steps in age hardening treatment of the alloy composite after choosing a proper composition of alloy: --

1. *Solutionising:* heating the alloy to a temperature at about 540°C to obtain a single-phase solid solution α.
2. *Quenching:* the solutionised alloy is cooled rapidly to retain the high temperature single-phase supersaturated solid solution at room temperature.
3. *Ageing:* age hardening by holding the quenched alloy at elevated temperature 180°C.

The hardness measurements were done by Vickers microhardness tester. Five sets of readings taken for each sample for better accuracy.

The thermal cycling of the composite is done by repeatedly solutionising the composite and taking the hardness measurements each time for a number of eight cycles.

Chapter 4

RESULTS AND DISCUSSION

4.1 Characterization of Zircon Particles

Scanning electron micrographs of zircon particles of various average particle size 65 μm are shown in Fig. 10. It shows that coarser particles are more spherical in shape compared to the finer ones.

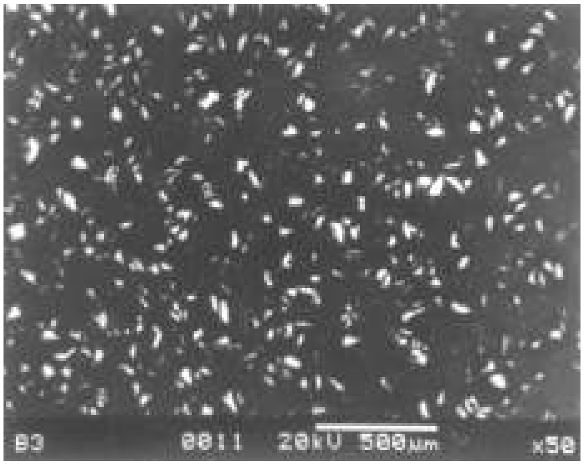


Fig. 10 - Scanning electron micrographs of the composite showing uniform **distribution of zircon sand particles**

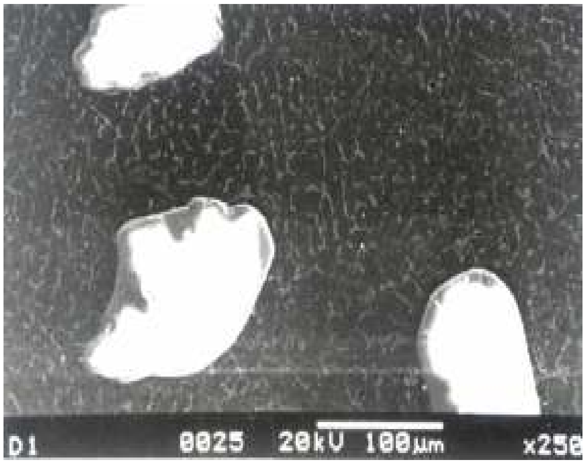


Fig. 11 - Scanning electron micrographs showing good particle matrix bonding

4.2 Microstructural examination of Composite

Scanning electron micrographs at low magnification show uniformly distributed zircon particles throughout the matrix (Fig.10). Scanning electron micrograph of as-cast composite at high magnification shows the presence of uniformly distributed secondary phase in the alloy matrix (Fig.11). There is no evidence of voids at the particle matrix interface, indicating a strong continuous bond between zircon particle and the alloy matrix.

X-ray diffraction pattern of as cast composite shows the presence of Al, $CuAl_2$ and zircon sand (Fig. 12). This confirms the presence of zircon particles and $CuAl_2$ phase in the cast composite. There is no crystallographic change in the tetragonal zircon during the synthesis process, confirming the stability of zircon particles.

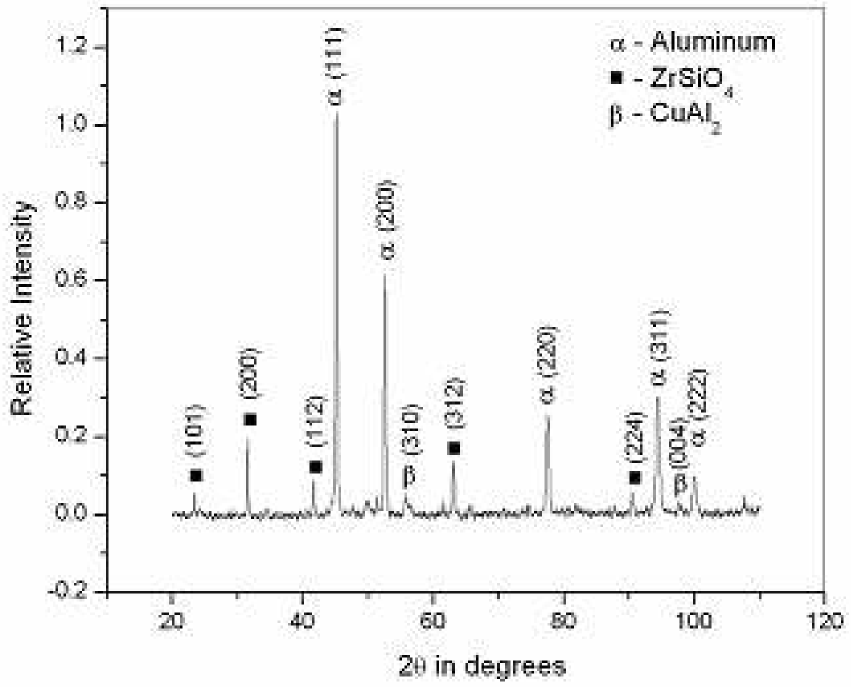


Fig.12 Typical X-Ray diffraction pattern for as cast composite

Optical micrograph of the as-cast alloy is shown in Fig. 13. The matrix of the composite has the cellular structure, where the size of the cell depends on the zircon particle size and amount in the composite

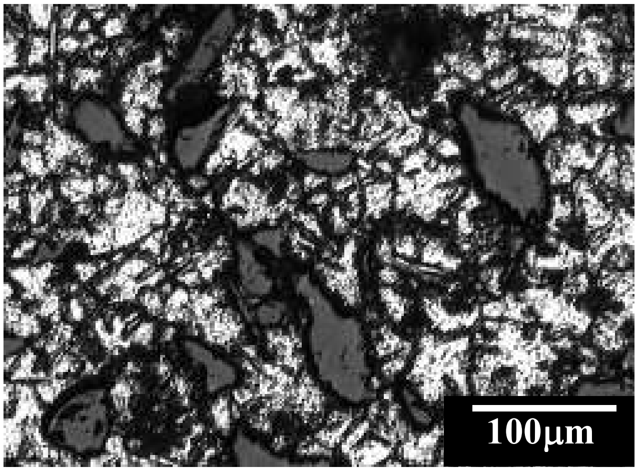


Fig. 13 - Optical microstructures of as cast composite

A polished metallography sample reveals circular grain type morphology of dendrites. In the present investigation, monolithic alloy shows dendritic structure. . In general there appears to be a reasonably uniform distribution of zircon sand particles. (Fig. 13)

4.3 Ageing Studies of the composite

The Hardness measurements were done by Vicker's microhardness tester at a load 25g and for a dwell time of 20 seconds. A set of five readings is taken for better accuracy

Table 5-Ageing data of the composite in Water

Ageing Time (hrs)	Microhardness Hv					Mean Hv
0	65.7	67.1	66.0	66.4	66.6	66.36
2	66.0	67.5	66.7	65.7	65.8	66.34
4	66.6	71.0	70.8	69.2	68.6	69.24
6	68.17	70.03	66.6	67.3	68.6	68.14
8	68.6	70.0	71.0	66.0	72.6	69.64
10	70.8	66.0	70.44	69.09	69.4	69.14

Table 6-Ageing data of the composite in Oil

Ageing Time (hrs)	Microhardness Hv					Mean Hv
0	65.5	64.6	63.8	65.0	66.3	65.04
2	70.5	68.4	68.56	68.17	67.7	68.66
4	72.5	68.3	69.22	69.76	68.56	69.66
6	67.39	67.65	66.23	67.0	66.37	66.92
8	68.17	68.83	68.56	68.69	69.36	68072
10	69.23	69.76	68.04	66.5	67.5	68.20

Table 7-Ageing data of the composite in Salt brine solution

Ageing Time (hrs)	Microhardness Hv					Mean Hv
0	69.09	67.91	67.7	68.56	66.62	67.97

2	69.09	69.49	69.66	70.72	70.44	69.88
4	71.0	72.1	73.27	72.98	71.28	72.12
6	70.58	72.2	72.12	72.12	72.98	71.98
8	71.5	70.31	69.9	67.26	69.96	69.78
10	67.26	67.52	67.65	65.75	67.26	67.08

Ageing curves

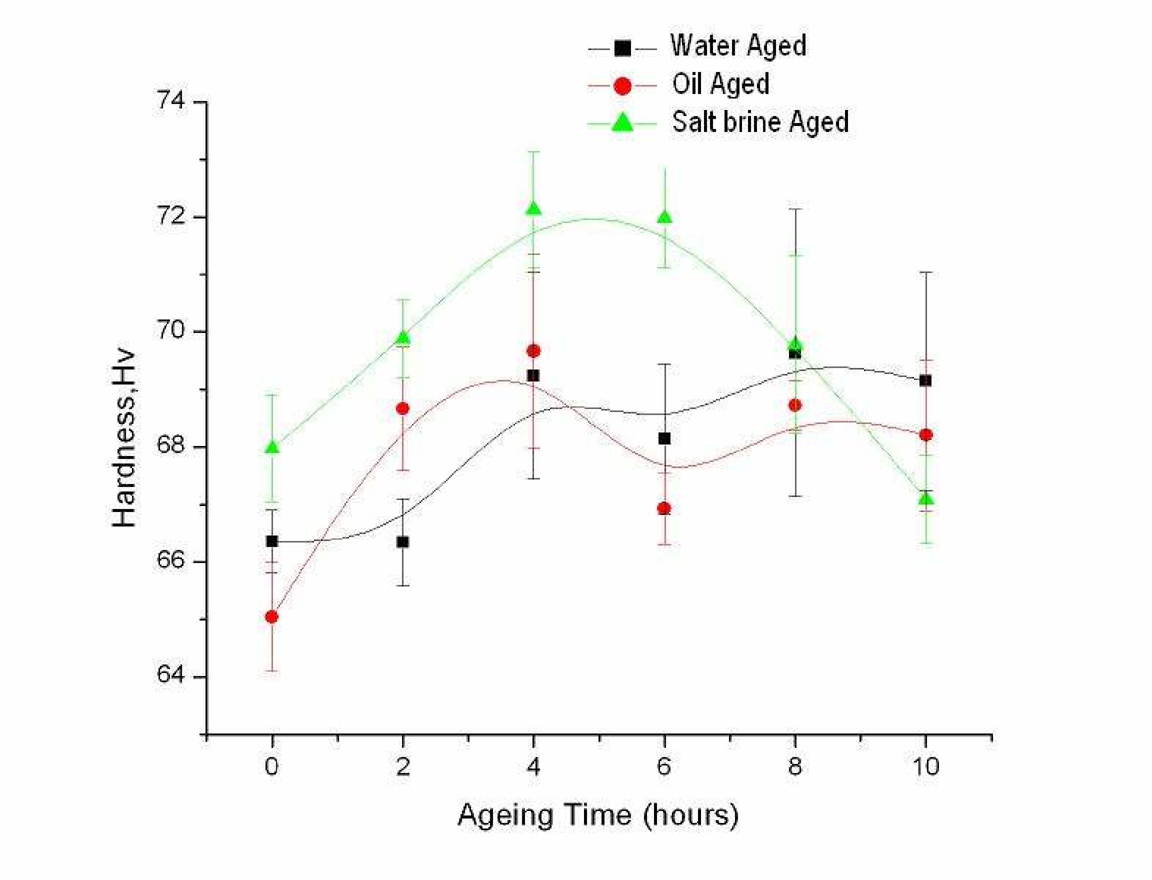


Fig. 14: Age hardening behavior of the composite in different quenching media

Table 8-Ageing Conditions

Solutionizing Temperature	540°C
Solutionizing Time (hour)	2 hours

Figure 14 shows the variation in microhardness as a function of ageing time for zircon sand particulate reinforced composite in different quenching media water, oil and salt bath. Water quenched samples takes longer time to attain the peak hardness values subjected to the ageing treatment. More dislocations are generated at the particle matrix interface due to their CTE mismatch as compared to the interparticle region. This causes copper atoms from the matrix to diffuse through the interfacial region. The concentration of Cu atoms in the matrix gradually decreases that's why the precipitation is delayed due to this copper leaned area and it shows peak hardness at larger ageing times. Here water quenched samples show peak hardness around 8 hrs in contradictory to oil quenched samples at around 4 hrs. Oil is classed as an intermediate quench. It has a slower cooling rate than brine or water. Distortion and danger of cracking is lower as compared to water or brine. Oil quenched sample show peak hardness at early stages of ageing, it is near 6 hrs as shown in Fig 14. This can be attributed to the presence of less dislocations after oil quenching, so that the copper atoms from the interparticle region does not diffuse significantly and their concentration is enough to cause early precipitation.

In salt brine quenched samples a large number dislocations are generated around the particle matrix interface, as it does not form the vapour blanket stage as in case of water. The time to peak hardness occurs in between water and salt brine quenched samples since the dislocation annihilation takes place after some time and the diffusion of copper atoms from the interparticle region to the particle matrix interface is not so much as in case of water quenched but more than oil quenched samples.

It will be clearer with the schematic illustration as shown in Fig 15

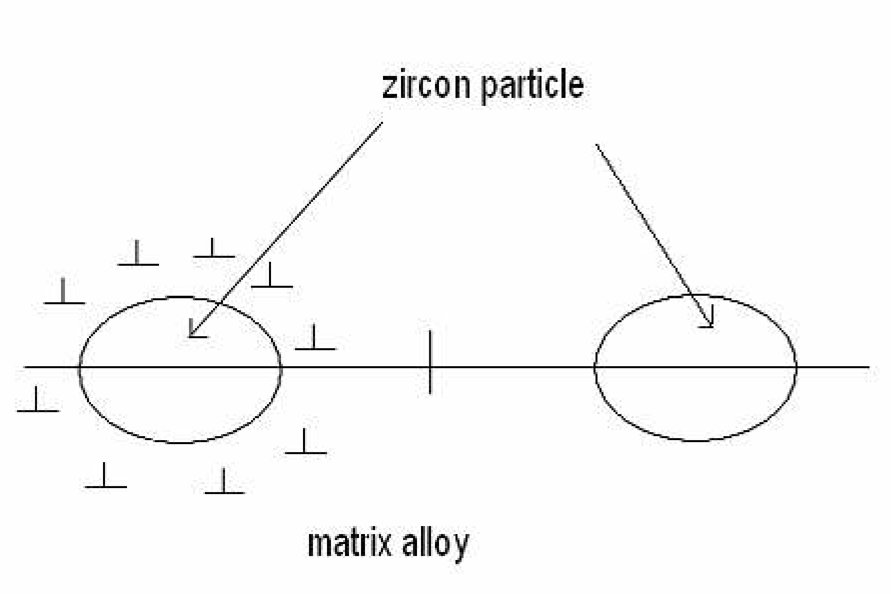


Fig.15 - Schematic diagram to show dislocations around the interparticle matrix interface

From Fig.15 initially the Cu solute atoms are dissolved uniformly in the matrix. The concentration of Cu atoms in matrix is more as compared to the particulate matrix bond region as a result, the diffusion/segregation of Cu atoms through this interfacial region and the enhanced dislocation density increases the hardness value as compared to the inter particle matrix region. Dislocations are more near interface as compared to interparticle region as shown but it is still more as compared to the monolithic alloy. That's why the composites usually show peak-aged condition much early as compared to monolithic alloy.

Under all the heat treatment conditions, the micro-hardness increases with the aging time to a peak value and then decreases with a prolonged aging time. This can be explained by the evolution of $CuAl_2$ precipitates with the aging time and the interaction between the precipitates and dislocations.

Salt solution has the advantage of providing more uniform quench over water by extending the vapor blanket stage to a lower temperature that's why more hardness obtained, but there is a danger of distortion and cracks in the sample so it is not commonly used for thin sections.

4.4 Thermal Cycling

Thermal cycling of the composite is done by solutionising repeatedly and taking the hardness meaurement each time.

Table 9-Thermal cycling data of the composite in Water

No. of Thermal Cycles	Microhardness Hv					Mean Hv
1	65.7	67.1	66.0	66.4	66.6	66.36
2	71.5	71.56	72.4	72.9	73.4	72.35
3	75.66	73.8	72.6	70.8	75.97	73.76
4	69.7	72.4	69.76	72.1	71.0	70.99
5	69.36	69.9	70.03	66.7	66.0	68.39
6	67.6	66.88	66.7	69.05	67	67.44
7	66.37	66.5	65.5	65.38	65.75	65.9
8	65.5	64.23	64.2	65.3	64.8	64.8

Table 10-Thermal cycling data of the composite in Oil

No. of Thermal Cycles	Microhardness Hv					Mean Hv
1	65.5	64.6	63.8	65.0	66.3	65.0
2	69.36	64.4	65.75	66.37	64.53	66.08
3	65.8	62.1	61.7	66.0	65.26	64.17
4	64.6	63.63	65.03	64.05	65.0	64.46
5	64.2	63.69	65.13	62.64	64.29	63.99
6	64.5	63.5	62.87	63.8	65.75	64.08
7	64.2	61.17	61.85	64.77	65.6	63.51
8	62.4	60.51	61.85	62.53	61.51	61.76

Table 11-Thermal cycling data of the composite in Salt brine solution

No. of Thermal Cycles	Microhardness Hv					Mean Hv
1	69.09	67.91	67.7	68.56	71.42	68.93
2	69.3	72.1	71.0	68.04	67.52	69.59
3	66.6	66.7	66.78	66.88	66.0	66.79
4	66.0	65.87	66.0	63.58	64.17	65.12
5	62.3	66.88	62.64	62.76	63.11	63.53
6	61.4	62.8	62.41	62.5	65.75	62.35
7	64.89	63.46	61.2	62.41	62.99	62.99
8	62.64	62.87	62.99	63.81	62.99	63.06

Thermal cycling Curves

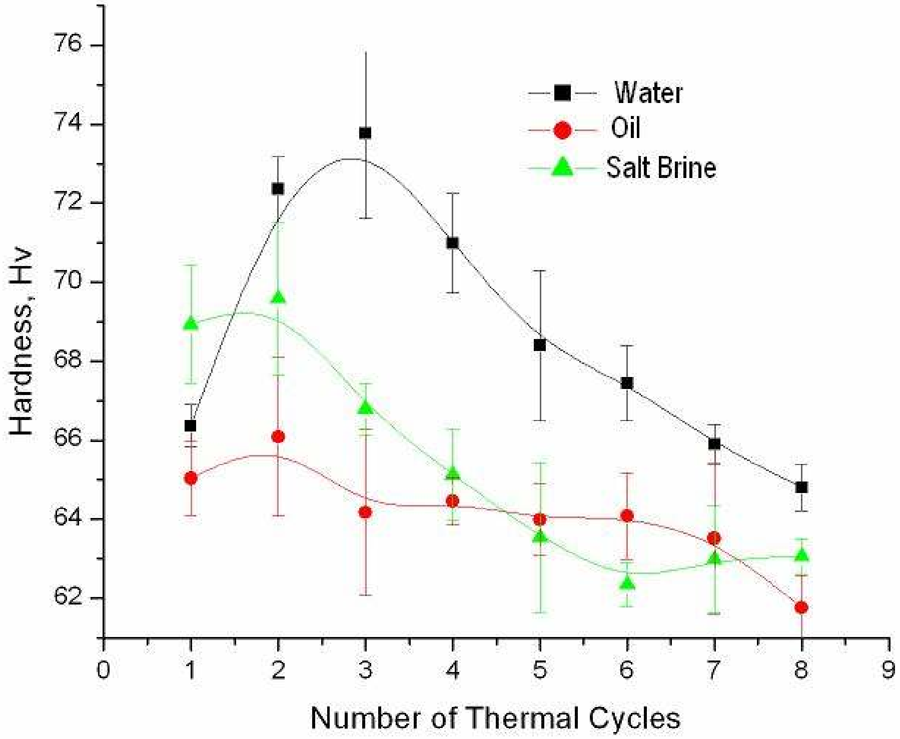


Fig.16 - Thermal cycling of the composite in different quenching media

The thermal cycling curve of this alloy composite has two distinct stages: the first stage is a rapid hardness increase in initial stages of thermal cycling observed until the maximum hardness occurs. (Figure 16). After this rapid reaction, the hardness curve exhibits a gradual drop with increasing number of thermal cycles.

When the specimen is heat-treated and quenched a large number of dislocation are generated due to the difference in CTE between the particulates and matrix. During initial thermal cycles the dislocation density gets enhanced drastically causing the rapid hardening. After a fixed number of thermally cycling the composite eventually a state is reached where the dislocations cancel each other due to the reduced interparticle spacing among themselves and forming a loop structure. In other words they annihilate and show the drop in hardness responsible for the plateau kind of stage in the graph.

After continuously thermally cycling the composite the hardness values drop as less dislocations are now generated. And after one or more thermal cycles the interface starts to crack under the thermally induced stress, it is more in salt brine.

If there is a strong interaction between the dislocation and the segregated solute atoms, dislocations become locked and hardening would precede the heterogeneous precipitation.

Water quenched samples produces more dislocations than oil quenched samples, while salt brine quenched samples attain the maximum hardness due to large number of dislocations as it does not form any kind of vapor blanket as in case of water.
Oil is less severe quenching medium; it produces less stresses hence hardness is minimum.

Chapter 5

SUMMARY AND CONCLUSIONS

- Zircon particles can be uniformly dispersed in Al-4.5wt% Cu alloy by the casting route.
- Under all the heat treatment conditions, the micro-hardness increased with the aging time to a peak value and then decreased with a prolonged aging time.
- Water quenched samples showed the peak hardness at later stages of ageing time, this can be attributed to the diffusion of copper atoms from the matrix to the interface region of the composite thus reducing the copper concentration in the interparticle region required for precipitation.
- Oil quenched samples showed the peak hardness in early stages of ageing hardness while salt brine quenched samples showed time to attain peak hardness in between water and oil quenched samples.
- Thermal cycling of the composite reveals that the microhardness of the composite increases with repeated solution heat treatment, attains a maximum and gradually drops with increasing number of heat treatment cycles. All the samples in different quenching media show a similar kind of behavior.
- Initial stage hardening is due to the increased number of dislocation during first few thermal cycles, and hardness drops at a later stage where the dislocations interact among themselves and annihilate each other with increasing the number of thermal cycles. Continuously thermally cycling causes the particle matrix interface to crack under the thermally induced stress. Cracking and distortion is more in case of salt brine solution.

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[70] The Aluminum Association, Inc. 900 19th Street, N.W. Washington, D.C. 20006
(202) 862-5100 www.aluminum.org