

# Study of Microstructure and Ageing Characteristic of Aluminium Alloy Reinforced with Glass Particulates

Ву

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# Glossary

Ageing hardening	=	A special dispersion-strengthening heat treatment. By solution treatment, quenching and ageing. A coherent precipitate forms that provides a substantial strengthening effect.
Discontinuous reinforced	=	Reinforced which is particle or whisker shapes
Endothermic	=	Endothermic reaction is one reaction that absorb energy/heat.
Eutectic	=	A three phase invariant reaction in which one liquid phase solidifies to produce two phases
Exothermic	=	Exothermic reaction is one reaction that release energy/heat.
GP zones	=	Guinier-Preston zone is tinny cluster of atom that precipitate from the matrix in the early stages of the age hardening process.
Hardness (test)	=	Measures the resistance of material to penetration by sharp object. Common hardness test include the Brinnel test, Rockwel test, Knop Test and Vickers test.
Matrix	X E	The continuous solid phase in a complex microstructure. Solid dispersed phase particles may form within the matrix.
Particulate	=	The materials powder that have more than one shape of particles.
Precipitation formation	=	A solid phase that forms from the original matrix phase when solubility limit is exceeded.
Quenching	=	Rapidly cooled after the solution heat treatment.
Reflectivity	=	The percentage of incident radiation that is reflected.
Silicide		Compound of silicon that have more electropositive
Solubility	=	The amount of one material that will completely dissolve in a second material without creating a second phase.
Solutionising	=	The first step of the precipitation strengthening heat treatment.
Thermal conductivity	=	A microstructure-sensitive property that measures the rate at which heat is transferred through material.

#### LIST OF ABBREVIATIONS, SYMBOLS and SPECIALISED **NOMENCLATURES**

ΑI **Aluminium** 

Al<sub>2</sub>Cu Aluminium Cupri

 $Al_2O_3$ Alumina

ASM American Society for Metals

oficinal copyright **ASTM** American Standard for Testing Materials

Be Beryllium С Composition CaO Calcium oxide

Cd Cadmium CI Chlorine Co Cobalt Cr Chromium

CTE Coefficient Thermal Expansion

Cu Cuprum D Diameter

DSC Differential Scanning Calorimetry

**EDS Energy Dispersive Spectrum** 

F Fluorine Fe Ferro/Iron Feri oxide  $Fe_2O_3$ Ga Gallium Germanium Ge

Guinier-Preston

**GP** zones Guinier-Preston zone

Hardness Value

**ICDD** International Centre for Diffraction Data

**IPB** Interphase boundary

K Constant

 $K_2O$ Potassium oxide Potassium trioxide  $K_2O_3$ 

Mg Magnesium

MgO Magnesium oxide  $Mg_2Sn$ Mangan stransium

Milimeter mm

MMC Metal matrix composite

Natrium Na

Sodium oxide Na<sub>2</sub>O

Ni Nickel Pb Lead

P/M **Powder Metallurgy RPM** Round per minute

otiothal copyridations SEM Scanning Electron Microscope

Si Silicon SiC Silica

SiO<sub>2</sub> Silicon oxide

Sn Tin

SSS Supersaturated solid solution

Т Temperature

TiO<sub>2</sub> Titanium dioxide

٧ Vanadium

W-Mo Tungsten-Molybdenum

Weight wt

**XRD** X-ray diffraction

Zinc Zn

α Alpha

#### KAJIAN MIKROSTRUKTUR DAN SIFAT PENUAAN ALOI ALUMINIUM YANG DITETULANGI DENGAN ZARAH KACA

#### **ABSTRAK**

Kajian mikrostruktur dan sifat penuaan telah dilakukan pada aloi aluminium yang ditetulangi dengan zarah kaca. Komposit aluminium - 4 % berat kuprum dihasilkan dengan kaedah metalurgi serbuk. Kandungan 0, 5, 10, 15, 20 dan 25 % berat zarah kaca dicampurkan ke dalam aloi aluminium – 4 wt. % kuprum. Semua komposit dibuat dengan kaedah pembuatan meliputi : pencampuran, penekanan, dan pensinteran. Pensinteran dijalankan pada suhu 548° C. Penuaan komposit dilakukan dengan cara rawatan haba pada 510° C, diikuti dengan lindap kejut dengan cepat ke dalam air sejuk dan penuaan buatan selama 10 jam dalam suhu 160° C.

Analisis XRD dan DSC menunjukan pembentukan mendakan di dalam Al – 4 % berat Cu aloi dan komposit. Al – 4 % berat Cu aloi yang tidak ditetulangi menunjukkan mendakan Al $_2$ Cu semasa pensinteran, tetapi setelah perawatan larutan, Al $_2$ Cu dikesan mempunyai puncak yang kecil pada graf XRD. Hal ini menunjukkan bahawa Al $_2$ Cu tidak melarut dengan sempurna. Selanjutnya, semasa pensinteran, ada terjadinya mendakan Al $_2$ Cu pada Al – 4 % berat Cu aloi yang tidak ditetulangi dengan zarah kaca, tetapi setelah perawatan larutan, mendakan Al $_2$ Cu tidak larut telah diperhatikan pada antara muka zarah kaca dengan aluminium matrik. Hal ini telah menunjukkan bahawa Al $_2$ Cu tidak melarut dengan sempurna. Penuaan telah mempengaruhi pembentukan mendakan di dalam komposit kerana Al $_2$ Cu telah menunjukkan keamatan tertinggi pada graf XRD dibandingkan dengan komposit tanpa penuaan.

Mikrostruktur pada komposit menunjukkan bahawa zarah kaca tertabur dengan homogen di dalam matrik. Walaupun demikian zarah kaca pada kadar 10, 15, 20 dan 25 % berat mengasingkan diri dekat kuprum. Pembentukan fasa AlCu and Al<sub>2</sub>Cu telah dihalangi oleh kehadiran zarah kaca. Lapisan aluminium oksida di dalam matrik dihasilkan di antara aluminium, kuprum dan zarah kaca. Ianya lebih keras daripada matrik yang ditunjukkan dengan keretakan mikro pada lapisan aluminium oksida yang terjadi di antara muka lapisan aluminium oksida dengan aluminium. Selanjutnya, lapisan aluminium oksida meningkatkan kekerasan pada matrik dan bahawa ianya bertanggungjawab terhadap mekanisme kekerasan komposit.

Setelah pensinteran, nilai kekerasan menunjukkan bahawa komposit dengan kadar 25 % berat zarah kaca mempunyai nilai kekerasan tertinggi iaitu 34.87 HV daripada aloi aluminium tanpa tetulang iaitu 15.83 HV. Nilai kekerasan bertambah dengan penambahan kandungan zarah kaca di dalam aloi aluminium. Kinetik penuaan pada komposit lebih lambat daripada aloi aluminium yang tidak ditetulangi. Lambatnya kinetik penuaan dijangka kerana kehadiran zarah kaca yang mengganggu pembentukan mendakan aluminium-kuprum dan melambatkan pembentukan zon G.P..

# STUDY OF MICROSTRUCTURE AND AGEING CHARACTERISTIC OF ALUMINUIM ALLOY REINFORCED WITH GLASS PARTICULATES

#### **ABSTRACT**

Study on microstructure and ageing characteristics has been done on the aluminium alloy reinforced with glass particulates. Aluminium - 4 wt. % copper composites were produced by powder metallurgy technique. Composition of 0, 5, 10, 15, 20 and 25 wt. % glass particulates was mixed into aluminium - 4 wt. % copper alloy. All composites were fabricated by mixing, pressing and sintering. The sintering was performed at 548° C. Ageing of the composites was done by solution heat treatment at 510° C followed by quenched rapidly into cool water and artificial aged for 10 hours at 160° C.

 $\dot{X}RD$  and  $\dot{D}SC$  analysis showed the precipitation formation in unreinforced and reinforced AI - 4 wt. % Cu alloy. Unreinforced AI - 4 wt. % Cu alloy showed the precipitation of Al<sub>2</sub>Cu during sintering, but after solution treatment Al<sub>2</sub>Cu detected as small peak by  $\dot{X}RD$ . It is indicated that  $\dot{A}_{12}Cu$  did not completely dissolved. In addition, reinforced AI - 4 wt. % Cu alloy with glass particulates showed precipitation of  $\dot{A}_{12}Cu$  during sintering but after solution treatment, undissolved precipitates were observed at the interface between glass particulates and the aluminium matrix. It showed incomplete dissolution. Ageing has influenced the precipitation formation in the composite because  $\dot{A}_{12}Cu$  indicated higher intensity of  $\dot{X}RD$  pattern as compared to the composite without ageing.

The microstructures of the composites showed that the glass particulates were homogenously distributed in the matrix. However, glass particulates with the composition of 10, 15, 20 and 25 wt. % were found segregated near copper. The formation of AlCu and Al<sub>2</sub>Cu phases was interfered by the presence of glass particulates. The aluminium oxide layer in the matrix is produced between aluminium, copper and glass particulate. It is harder than matrix that indicated by micro cracks in aluminium oxide layer and at interface between aluminium oxide layer and aluminium. Furthermore, it was found an increase in the hardness of matrix and that is found responsible for hardening mechanism.

After sintering, hardness value indicated that the composite with 25 wt. % glass had highest hardness i.e. 34.87 HV than unreinforced aluminum alloy i.e. 15.83 HV. The hardness value increased with increasing the glass composition in the aluminium alloy. Ageing kinetic of the composites was slower than unreinfoced aluminium alloy. The slow ageing kinetic was expected due to the presence of glass particulates which disturbed precipitate formation of aluminium-copper and delay the G.P. zone formation.

#### **CHAPTER 1**

#### INTRODUCTION

#### 1.1. Research background

Production of aluminium is the most abundant metallic element in the earth's crust and it is ranked only second to iron and steel in the metals market. The rapid growth of the aluminium industry is attributed to a unique combination of properties which makes it one of the most versatile of engineering and construction materials. Aluminium can be cast and worked into almost any form and can be given a wide variety of surfaces finishes (Smith, 1993).

Aluminium is light in weight, some of its alloys have good strength, and it has good electrical and thermal conductivities and high reflectivity to both heat and light. It is highly corrosion-resistance under a great many service condition and its non toxic (Smith, 1993, Kissell and Ferry, 2002). Because of these outstanding properties, it is not surprising that aluminium alloys have come to be of prime importance as engineering materials, one of it is aluminium - copper alloy.

Copper is one of most important alloying elements for aluminium since it produces considerable solid solution strengthening that provide greatly increased strength by precipitate formation. The maximum solid solubility of copper in aluminium is 5.65 wt. % at the eutectic temperature of 548°C. The solubility of copper in aluminium decreases rapidly with decreasing temperature from 5.65 wt. % Cu to less than about 0.1 wt. % Cu at temperature room as shown in Figure 1.1 (Smith, 1993).

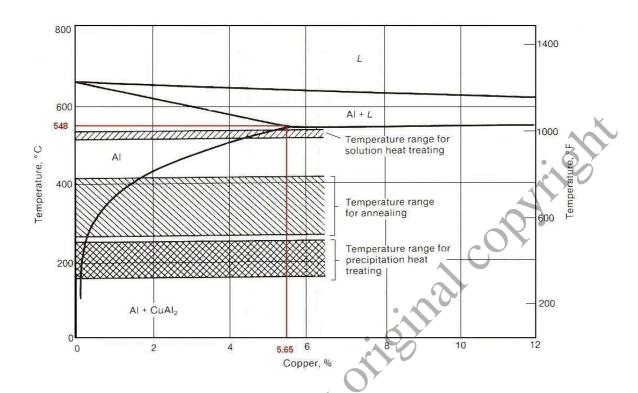


Figure 1.1. Phase diagram Al-Cu Alloy (ASM Handbook, 1991).

Discontinuously (particles) reinforced aluminium matrix composites have received significant attention in recent years. The high specific strength is like hardness make discontinuous reinforced aluminium composites attractive as candidate materials for many applications in industry (KK. Chawla et, al., 1972). The great number of glasses available from recycling activity of industrial wastes leads to the need for new applications, with the development of new materials such as low cost composite materials from a powder technology route (E. Bernardo, et. al, 2004).

#### 1.2. Problem statement

In this research, aluminium-copper alloys as prime importance as materials engineering is treated as composite and it is added with glass as reinforcement. Commercially source of glass is abundance and glass disposal can be recycled as particulate reinforcement in metal matrix composite.

Glass is chosen because it is popular as reinforcement materials because it is easily drawn into high-strength fibre from the molten state (Calijster, 2003). The fact of the glass is relatively weak in tension but relatively strong in compression (Shackelford, 2005). In some general statements it is made concerning to the properties of glass is harder than many metals (400 to 600 kg/mm<sup>2</sup>) and glasses have a compressive strength of about 945 MPa (Horrat, 2001). ्ती <sup>ट</sup>०१

#### 1.3. Objectives of the research

The objectives of this research are:

- 1. To fabricate aluminium-copper both of reinforced and unreinforced with recycled window glass by powder metallurgy technique.
- 2. To carry out heat treatment ageing to the composite to improve the hardness property.
- 3. To investigate its microstructure and to study the effect of glass addition on ageing characteristic

#### 1.4. Scope of the research

Fabrication of aluminium matrix composite is carried out by powder metallurgy technique. Powder metallurgy is well established for the production of discontinuous fibre, whiskers or particulate reinforced metals. The components are mixed and then pressed, followed by sintering at high temperature (Matthews and Rawling. 2002). Flow chart of the research methodology is given in Figure 1.2.

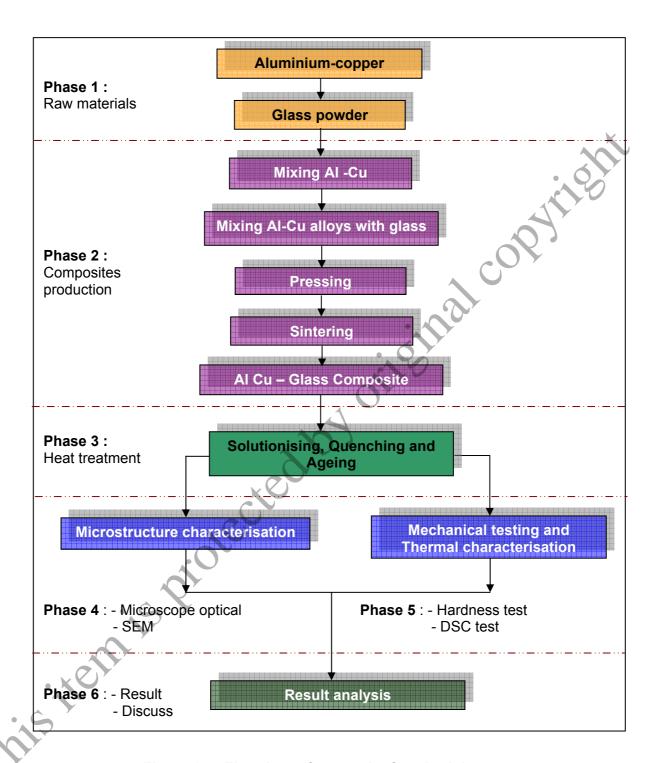


Figure 1.2. Flowchart of research of methodology.

In this methodology is used technical term of research flow by Phase 1, 2, 3, 4, 5 and 6 while each phase is representative of level work.

Phase 1 in Figure 1.2 shows that raw material should be provided especially for glass particulates. Glass particulates obtained from glass disposal and processed to be

particulate. The size glass particulates as reinforcement must be smaller than aluminium matrix so, that ease drawn into matrix.

In technical term of Phase 2 is process to fabricate the composites that shown which is mix the aluminium with copper and then Al-Cu mix with glass particulates. In powder metallurgy technique after mixed the materials powder will go to press then sinter to obtain product of composites. Phase 3 is treating of sample to prepare the data of the composites. In this research that the way to get the hardness property of aluminium-copper composites are solutionising, quenching and ageing.

Term of Phase 4 explained that Microscope optical and Scanning Electron Microscope (SEM) to carry out the composites microstructure characterisation.

In this research, the thermal characterisation is used Differential Scanning Calorimetric (DSC) aiming to explore if the copper is present in solid solution and the glass presence to influence the process solutionising. It is related to the ageing and the hardness property of composites (term of Phase 5). Phase 6 shows discussion can be done if all results of data have been completed.

#### **CHAPTER 2**

## LITERATURE REVIEW

#### 2.1. Introduction

The concept of composites was not invented by human being, it is found in nature. For an example, wood which is a natural composite material consisting of one species of polymer called cellulose fibres with good strength and stiffness in resinous matrix of another polymer called the polysaccharide lignin. In addition, celery, bamboo, corn, bone, teeth and mollusk are all examples of nature's composite. Other common variety of composites made by human being, an example in India, Greece and other countries, husks, sawdust or straw mixed with clay have been used to build houses for several hundred years. Mixing husk or sawdust in clay is an example of particulate composite and mixing straws in clay is an example of short fibre composite (Harris, 1999, Horat, 2001, Mazumdar, 2002).

The main concept of composite is that it contains matrix material. Typically, composite material is formed by reinforcing fibre in matrix resin as shown in Figure 2.1. The reinforcements can be fibres, particulates, or whiskers, and matrix materials can be metals, polymers or ceramics (Mazumdar, 2002).

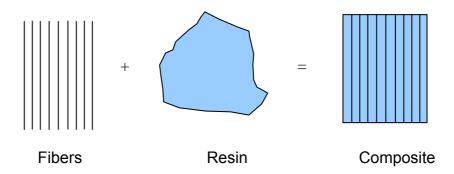


Figure 2.1. Formation of composite material using fibres and resin (Mazumdar, 2002).

According to Mazumdar (2002), reinforcement of composite has function to carry the load in structural composites, for the fibre carries 70 % to 90 % of the load. The reinforcement of composite also provides stiffness, strength, thermal stability, and other structural properties in the composites. Electrical conductivity or insulation has provided, depending on the type of reinforcement used.

A matrix of materials fulfils several functions in a composite structure, most of which are vital to the satisfactory performance of the satisfactory performance of the structure. The important function of matrix material binds the fibres together and transfers the load to the fibres. It provides rigidity and shape to the structure. The matrix isolates the fibers so that individual fibres can act separately. This stops or slows the propagation of a crack. The matrix provides a good surface finish quality and aids in the production of net-shape or near-net-shape parts. The matrix provides protection to reinforcing fibres against chemical attack and mechanical damage (wear). Depending in the matrix material selected, performance characteristics such as ductility, impact strength, are also influenced. A ductile matrix will increase the toughness of the structure. For higher toughness requirements, thermoplastic-based composites are selected. The failure mode is strongly affected by the type of matrix material used in the composite as well as its compatibility with the fibre.

Composite materials can be selected to give unusual combinations of stiffness, strength, weight, high-temperature performance, corrosion resistance, conductivity or hardness (Askeland and Phule, 2006).

In general a composite is a material made by combining two or more materials to give a unique combination of properties that exhibit improved properties over their individual components (Harris, 1999, Horat, 2001, Mazumdar, 2002, Calijster, 2003).

#### 2.2. Strengthening mechanism in metals

Strengthening mechanisms is the relation between dislocation motion and mechanical behaviour of metals. Because macroscopic plastic deformation corresponds to the motion of large numbers of dislocation, the ability of a metal to plastically deform depends on the ability of dislocations to move. Since hardness and strength (both yield and tensile) are related to the ease with plastic deformation can be made to occur, by reducing the mobility of dislocations, the mechanical strength can be enhanced; that is, greater mechanical forces will be required to initiate plastic deformation. In contrast the more unconstrained the dislocation motion, the greater the facility with which a metal deform, and the softer and weaker it becomes. Virtually all strengthening techniques rely on this simple principle; restricting or hindering dislocation motion renders a material harder and stronger (Calijster, 2003).

Strengthening mechanisms are as following: solid solution strengthening, strain or work hardening, grain boundary strengthening, dispersion strengthening and precipitation hardening (Henkel & Pense, 2001, Calijster, 2003, Bowman, 2004).

#### 2.2.1. Solid solution strengthening

Solid solution is a solid containing two or more elements atomically dispersed at random in single crystalline structure. In the substitutional type of solid solution, atoms of the solute element are substituted for solvent atoms at random points in its lattice. In