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Manufacturing and Properties of Quartz (SiO$_2$) Particulate Reinforced Al-11.8%Si Matrix Composites

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Additional information is available at the end of the chapter

1. Introduction

Metal matrix composites (MMC) are a class of composites that contains an element or alloy matrix in which a second phase is fixed firmly deeply and distributed evenly to achieve the required property improvement. The property of the composite varies based on the size, shape and amount of the second phase (Sayuti et al., 2010; Sulaiman et al., 2008). Discontinuously reinforced metal matrix composites, the other name for particulate reinforced composites, constitute 5 – 20 % of the new advanced materials (Gay et al., 2003). The mechanical properties of the processed composites are greatly influenced by their microstructure. An increased stiffness, yield strength and ultimate tensile strength are generally achieved by increasing the weight fraction of the reinforcement phase in the matrix. Inspite of these advantages, the usage of particulate reinforced MMCs as structural components in some applications is limited due to low ductility (Rizkalla and Abdulwahed, 1996). Owing to this and to overcome the draw-backs, a detailed investigation on the strengthening mechanism of composites has been carried out by composite experts (Humphreys, 1987). They have found that the particle size and its weight fraction in metal matrix composites influences the generation of dislocations due to thermal mismatch. The effect is also influenced by the developed residual and internal stresses too. The researchers have predicted that the dislocation density is directly proportional to the weight fraction and due to the amount of thermal mismatch. As a result, the strengthening effect is proportional to the square root of the dislocation density. This effect would be significant for fine particles and for higher weight fractions. The MMCs yield improved physical and mechanical properties and these outstanding benefits are due to the combined metallic and ceramic properties (Hashim et al., 2002). Though there are various types of MMCs, particulate-reinforced composites are the most

versatile and economical (Sayuti, Sulaiman, Vijayaram, et al., 2011; Sayuti, Suraya, et al., 2011).

In the past 40 years, the researchers and design experts have perceived their research to emphasis on finding lightweight, environmental friendly, low-cost, high quality, and good performance materials (Feest, 1986). In accordance with this trend, MMCs have been attracting growing interest among researchers and industrialists. The attributes of MMCs include alterations in mechanical behavior (e.g., tensile and compressive properties, creep, notch resistance, and tribology) and physical properties (e.g., intermediate density, thermal expansion, and thermal diffusivity) a change, primarily induced by the reinforced filler phase (Sayuti, et al., 2011). Even though MMCs posses various advantages, they still have limitations of thermal fatigue, thermo-chemical compatibility, and posses lower transverse creep resistance. In order to overcome these limitations, fabrication of discontinuously reinforced Al-based MMCs was carried out by standard metallurgical processing methods such as powder metallurgy, direct casting, rolling, forging and extrusion. Subsequently, the products were shaped, machined and drilled by using conventional machining processes. Consequently, the MMCs would be available in suitable quantities with desirable properties, particularly for automotive applications (Sharma et al., 1997).

In general, composite materials posses good mechanical and thermal properties, sustainable over a wide range of temperatures (Vijayaram et al., 2006). The desirable factors such as property requirements, cost factor considerations and future application prospects would decide the choice of the processing method (Kaczmar et al., 2000). In practice, composite materials with a metal or an alloy matrix are fabricated either by casting or by powder metallurgy methods (Fridlyander, 1995). They are considered as potential material candidates for a wide variety of structural applications in the transportation, automobile and sport goods manufacturing industries due to the superior range of mechanical properties they exhibit (Hashim et al., 1999). MMCs represent a new generation of engineering materials in which a strong ceramic reinforcement is incorporated into a metal matrix to improve its properties such as specific strength, specific stiffness, wear resistance, corrosion resistance and elastic modulus (Baker et al., 1987; Chambers et al., 1996; Kok, 2005). As a virtue of their structure and bonding between the matrix and the reinforcement, MMCs combine metallic properties of matrix alloys (ductility and toughness) with ceramic properties of reinforcements (high strength and high modulus), therein leading to greater strength in shear and compression as well as higher service-temperature capabilities (Huda et al., 1993). Thus, they have scientific, technological and commercial significance. MMCs, because of their improved properties, are being used extensively for high performance applications such as in aircraft engines especially in the last decade. Recently, they also find application in automotive sectors (Surappa, 2003; Therén and Lundin, 1990).

Aluminum oxide (Al_2O_3) and silicon carbide (SiC) powders in the form of fibers and particulates are commonly used as reinforcements in MMCs. In the automotive and aircraft industries for example, production of engine pistons and cylinder heads, the tribological properties of the materials used are considered crucial. Hence, Aluminum oxide and silicon carbide reinforced aluminum alloy matrix composites are applied in these fields (Prasad and

Asthana, 2004). Due to their high demand, the development of aluminum matrix composites is receiving considerable emphasis in modern application. Research reports ascertain that the incorporation of hard second phase particles in the alloy matrices to produce MMCs is beneficial and economical due to its high specific strength and corrosion resistance properties (Kok, 2005). Therefore, MMCs are those materials that have higher potential for a large range of engineering applications.

2. Metal Matrix Composites (MMCs)

Metal matrix composites are a family of new materials which are attracting considerable industrial interest and investment worldwide. They are defined as materials whose microstructures compromise a continuous metallic phase (the matrix) into which a second phase, or phases, have been artificially introduced. This is in contrast to conventional alloys whose microstructures are produced during processing by naturally occurring phase transformations (Feest, 1986). Metal matrix composites are distinguished from the more extensively developed resin matrix composites by virtue of their metallic nature in terms of physical and mechanical properties and by their ability to lend themselves to conventional metallurgical processing operations. Electrical conductivity, thermal conductivity and non-inflammability, matrix shear strength, ductility (providing a crack blunting mechanism) and abrasion resistance, ability to be coated, joined, formed and heat treated are some of the properties that differentiate metal matrix composites from resin matrix composites. MMCs are a class of advanced materials which have been developed for weight-critical applications in the aerospace industry. Discontinuously reinforced aluminum composites, composed of high strength aluminum alloys reinforced with silicon carbide particles or whiskers, are a subclass of MMCs. Their combination of superior properties and fabricability makes them attractive candidates for many structural components requiring high stiffness, high strength and low weight. Since the reinforcement is discontinuous, discontinuously reinforced composites can be made with properties that are isotropic in three dimensions or in a plane. Conventional secondary fabrication methods can be used to produce a wide range of composites products, making them relatively inexpensive compared to the other advanced composites reinforced with continuous filaments. The benefit of using composite materials and the cause of their increasing adoption is to be looked for in the advantage of attaining property combinations that can result in a number of service benefits. Among these are increased strength, decreased weight, higher service temperature, improved wear resistance and higher elastic module. The main advantage of composites lies in the tailorability of their mechanical and physical properties to meet specific design criteria. Composite materials are continuously displacing traditional engineering materials because of their advantages of high stiffness and strength over homogeneous material formulations. The type, shape and spatial arrangement of the reinforcing phase in metal matrix composites are key parameters in determining their mechanical behavior. The hard ceramic component which increases the mechanical characteristics of metal matrix composites causes quick wear and premature tool failure in the machining operations. Metal matrix composites have been investigated since the early 1960s with the impetus at that time, being the high potential structural properties

that would be achievable with materials engineered to specific applications (Mortensen et al., 1989).

In the processing of metal matrix composites, one of the subjects of interest is to choose a suitable matrix and a reinforcement material (Ashby and Jones, 1980). In some cases, chemical reactions that occur at the interface between the matrix and its reinforcement materials have been considered harmful to the final mechanical properties and are usually avoided. Sometimes, the interfacial reactions are intentionally induced, because the new layer formed at the interface acts as a strong bond between the phases (Gregolin et al., 2002).

During the production of metal matrix composites, several oxides have been used as reinforcements, in the form of particulates, fibers or as whiskers (Zhu and Iizuka, 2003). For example, alumina, zirconium oxide and thorium oxide particulates are used as reinforcements in aluminum, magnesium and other metallic matrices (Upadhyaya, 1990). Very few researchers have reported on the use of quartz as a secondary phase reinforcement particulate in an aluminum or aluminum alloy matrix, due to its aggressive reactivity between these materials (Sahin, 2003). Preliminary studies showed that the contact between molten aluminum and silica-based ceramic particulates have destroyed completely the second phase microstructure, due to the reduction reaction which provokes the infiltration of liquid metal phase into the ceramic material (Mazumdar, 2002). Previous works carried out by using continuous silica fibers as reinforcement phases in aluminum matrix showed that even at temperatures nearer to 400 ^{0}C, silica and aluminum can react and produce a transformed layer on the original fiber surface as a result of solid diffusion between the phases and due to the aluminum-silicon liquid phase formation (Seah et al., 2003). The organizations and companies that are very active in the usage of MMCs in Canada and United States include the following (Rohatgi, 1993):

1. Aluminium Company of Canada, Dural Corporation, Kaiser Aluminium, Alcoa, American Matrix, Lanxide, American Refractory Corporation
2. Northrup Corporation, McDonald Douglas, Allied Signal, Advanced Composite Materials Corporation, Textron Specialty Materials
3. DWA Associates, MCI Corporation, Novamet
4. Martin Marietta Aerospace, Oakridge National Laboratory , North American Rockwell, General Dynamics Corporation, Lockheed Aeronautical Systems
5. Dupont, General Motors Corporation, Ford Motor Company, Chrysler Corporation, Boeing Aerospace Company, General Electric , Westinghouse
6. Wright Patterson Air Force Base, (Dayton, Ohio), and
7. Naval Surface Warefare Centre, (Silver Spring, Maryland)

India also has substantial activity in PM and cast MMCs. It has had world class R&D in cast aluminium particulate composites which was sought even by western countries.

2.1. Classification of composites

Among the major developments in materials in recent years are composite materials. In fact, composites are now one of the most important classes of engineered materials, because they

offer several outstanding properties as compared to conventional materials. The matrix material in a composite may be ceramic based, polymer or metal. Depending on the matrix, composite materials are classified as follows:

Metal matrix composites (MMCs)
Polymer matrix composites (PMCs)
Ceramic matrix composites (CMCs)

Majority of the composites used commercially are polymer-based matrices. However, metal matrix composites and ceramic matrix composites are attracting great interest in high temperature applications (Feest, 1986). Another class of composite material is based on the cement matrix. Because of their importance in civil engineering structures, considerable effort is being made to develop cement matrix composites with high resistance to cracking (Schey, 2000). Metal matrix composites (MMCs) are composites with a metal or alloy matrix. It has resistance to elevated temperatures, higher elastic modulus, ductility and higher toughness. The limitations are higher density and greater difficulty in processing parts. Matrix materials used in these composites are usually aluminum, magnesium, aluminum-lithium, titanium, copper and super alloys. Fiber materials used in MMCs are aluminum oxide, graphite, titanium carbide, silicon carbide, boron, tungsten and molybdenum. The tensile strengths of non metallic fibers range between 2000 MPa to 3000 MPa, with elastic modulus being in the range of 200 GPa to 400 GPa. Because of their lightweight, high specific stiffness and high thermal conductivity, boron fibers in an aluminum matrix have been used for structural tubular supports in the space shuttle orbiter. Metal matrix composites having silicon carbide fibers and a titanium matrix, are being used for the skin, stiffeners, beams and frames of the hypersonic aircrafts under development. Other applications are in bicycle frames and sporting goods (Wang et al., 2006). Graphite fibers reinforced in aluminum and magnesium matrices are applied in satellites, missiles and in helicopter structures. Lead matrix composites having graphite fibers are used to make storage-battery plates. Graphite fibers embedded in copper matrix are used to fabricate electrical contacts and bearings. Boron fibers in aluminum are used as compressor blades and structural supports. The same fibers in magnesium are used to make antenna structures. Titanium-boron fiber composites are used as jet-engine fan blades. Molybdenum and tungsten fibers are dispersed in cobalt-base super alloy matrices to make high temperature engine components. Squeeze cast MMCs generally have much better reinforcement distribution than compocast materials. This is due to the fact that a ceramic perform which is used to contain the desired weight fraction of reinforcement rigidly attached to one another so that movement is inhibited. Consequently, clumping and dendritic segregation are eliminated. Porosity is also minimized, since pressure is used to force the metal into interfiber channels, displacing the gases. Grain size and shape can vary throughout the infiltrated preform because of heat flow patterns. Secondary phases typically form at the fiber-matrix interface, since the lower freezing solute-rich regions diffuse toward the fiber ahead of the solidifying matrix (Surappa, 2003).

2.2. Significance of composites

Composites technology and science requires interaction of various disciplines such as structural analysis and design, mechanics of materials, materials science and process

engineering. The tasks of composites research are to investigate the basic characteristics of the constituents and composite materials, develop effective and efficient fabrication procedures, optimize the material for service conditions and understanding their effect on material properties and to determine material properties and predict the structural behavior by analytical procedures and hence to develop effective experimental techniques for material characterization, failure analysis and stress analysis (Daniel and Ishai, 1994). An important task is the non-destructive evaluation of material integrity, durability assessment, structural reliability, flaw criticality and life prediction. The structural designs and systems capable of operating at elevated temperatures has spurred intensive research in high temperature composites, such as ceramic/matrix, metal/ceramic and carbon/carbon composites. The utilization of conventional and new composite materials is intimately related to the development of fabrication methods. The manufacturing process is one of the most important stages in controlling the properties and ensuring the quality of the finished product. The technology of composites, although still developing, has reached a state of maturity. Nevertheless, prospects for the future are bright for a variety of reasons. Newer high volume applications, such as in the automotive industry, will expand the use of composites greatly.

2.3. Matrix

Matrix is the percolating alloy/metal/polymer/plastic/resin/ceramic forming the constituent of a composite in which other constituents are embedded. If the matrix is a metal, then it is called as a metal matrix and consecutively polymer matrix, if the matrix is a polymer and so on. In composites, the matrix or matrices have two important functions (Weeton et al., 1988). Firstly, it holds the reinforcement phase in the place. Then, under an applied force, it deforms and distributes the stress to the reinforcement constituents. Sometimes the matrix itself is a key strengthening element. This occurs in certain metal matrix composites. In other cases, a matrix may have to stand up to heat and cold. It may conduct or resist electricity, keep out moisture, or protect against corrosion. It may be chosen for its weight, ease of handling, or any of many other applications. Any solid that can be processed to embed and adherently grip a reinforcing phase is a potential matrix material.

In a composite, matrix is an important phase, which is defined as a continuous one. The important function of a matrix is to hold the reinforcement phase in its embedded place, which act as stress transfer points between the reinforcement and matrix and protect the reinforcement from adverse conditions (Clyne, 1996). It influences the mechanical properties, shear modulus and shear strength and its processing characteristics. Reinforcement phase is the principal load-carrying member in a composite. Therefore, the orientation, of the reinforcement phase decides the properties of the composite.

2.4. Reinforcing phase / Materials

Reinforcement materials must be available in sufficient quantities and at an economical rate. Recent researches are directed towards a wider variety of reinforcements for the range of

matrix materials being considered, since different reinforcement types and shapes have specific advantages in different matrices (Basavarajappa et al., 2004). It is to be noted that the composite properties depend not only on the properties of the constituents, but also on the chemical interaction between them and on the difference in their thermal expansion coefficients, which both depend on the processing route. In high temperature composites, the problem is more complicated due to enhanced chemical reactions and phase instability at both processing and application temperatures. Reinforcement phases in MMCs are embedded in the form of continuous reinforcement or discontinuous reinforcement in the matrix material. The reinforcing phase may be a particulate or a fiber, continuous type or discontinuous type. Some of the important particulates normally reinforced in composite materials are titanium carbide, tungsten carbide, silicon nitride, aluminum silicate, quartz, silicon carbide, graphite, fly ash, alumina, glass fibers, titanium boride etc. The reinforcement second phase material is selected depending on the application during the processing of composites (Clyne, 1996). The reinforcement phase is in the form of particulates and fibers generally. The size of the particulate is expressed in microns, micrometer. However, the discontinuous fiber is defined by a term called as 'Aspect Ratio'. It is expressed as the ratio of length to the diameter of the fiber. To improve the wettabilty with the liquid alloy or metal matrix material, the reinforcement phase is always preheated (Adams et al., 2003).

2.5. Factors affecting reinforcement

The interface between the matrix and the reinforcement plays an important role for deciding and explaining the toughening mechanism in the metal matrix composites. The interface between the matrix and the reinforcement should be organized in such a way that the bond in between the interface should not be either strong or weak (Singh et al., 2001).

2.6. Matrix interface / Interphase of matrices

Interfaces are considered particularly important in the mechanical behavior of MMCs since they control the load transfer between the matrix and the reinforcement. Their nature depends on the matrix composition, the nature of the reinforcement, the fabrication method and the thermal treatments of the composite. For particular matrix/reinforcement associations and especially with liquid processing routes, reactions can occur which change the composition of the matrix and lead to interfacial reaction products, thus changing the mechanical behavior of the composites. The interfacial phenomena in MMCs have been surveyed by several authors. Considering physical and chemical properties of both the matrix and the reinforcing material, the actual strength and toughness desired for the final MMCs, a compromise has to be achieved balancing often several conflicting requirements. A weak interface will lead to crack propagation following the interface, while a strong matrix associated with a strong interface will reveal cracks across both the matrix and the reinforcements. If however the matrix is weak in comparison with the interface and the particle strength, the failure will propagate through the matrix itself. The wettability of the

reinforcement material by the liquid metallic matrix plays a major role in the bond formation. It mainly depends on heat of formation, electronic structure of the reinforcement and the molten metal temperature, time, atmosphere, roughness and crystallography of the reinforcement. Similarity between metallic bond and covalent bond is reflected in some metal, like titanium carbide and zirconium carbide which are more easily wetted than strong ionic bonds found in ceramics such as alumina that remains poorly wetted. Surface roughness of the reinforced material improves the mechanical interlocking at the interface, though the contribution of the resulting interfacial shear strength is secondary compared to chemical bonding. Large differences in thermal expansion coefficient between the matrix and the reinforcement should be avoided as they can include internal matrix stresses and ultimately give rise to interfacial failures. From a purely thermo dynamical point of view, a comparison of free enthalpy of formation at various temperatures shows that many metals in the liquid state are reactive toward the reinforcing materials in particular oxides or carbides. Though thermodynamically favored, some reactions are however not observed and practically the kinetics of these reactions has to be considered in conjunction with thermodynamic data in order to evaluate the real potential of the reactions. The consequences of such interfacial reactions are the chemical degradation of the reinforcing material associated with a decrease of its mechanical properties, the formation of brittle reaction products at the interface, as well as the release of elements initially part of the reinforcing material toward the matrix may generate inopportune metallurgical phases at the vicinity of the reinforcing materials. Moreover in the case of alloyed matrices, the selective reactivity and depletion of given elements from the alloy can generate compositional gradients in the matrix and may therefore alter its properties close to the interface. Though a moderate reaction may improve the composite bonding, extended reactions usually ruin the reinforcing material. The relation between interfacial reactions and interface strength depends on the materials. The elaboration of MMC requires often a very short solidification time to avoid excess interfacial reaction. During the cooling process, differences in thermal capacity and thermal conductivity between the reinforcing material and the matrix induce localized temperature gradients. Solidification of the metallic matrix is believed to be generally a directional outward process, starting from the inside of the metallic matrix while ending at the reinforcing material surface. Finally, the processing type and the parameters have to be selected and adjusted to a particular MMC system. Metals are generally more reactive in the liquid rather than in the solid state. Consequently, shorter processing time, that is, short contact time between the liquid metal and the reinforcement can limit the extent of interfacial reactions. The study of reinforcement and matrix bonding is important in composite matrix structure, which has been described by Gregolin (2002). While the load is acting on the composite, it has been distributed to the matrix and the reinforcement phase through the matrix interface. The reinforcement is effective in strengthening the matrix only if a strong interfacial bond exists between them. The interfacial properties also influence the resistance to crack propagation in a composite and therefore its fracture toughness (Dusza and Sajgalik, 1995). The two most important energy-absorbing failure mechanisms in a composite are debonding and particle pull-out at the particle matrix interface. If the interface between the matrix and reinforcement debonds,

then the crack propagation is interrupted by the debonding process and instead of moving through the particle, the crack moves along the particle surface allowing the particle to carry a higher load (El-Mahallawy and Taha, 1993).

2.7. Physical phenomena of wettability and application

Wettability is defined as the extent to which a liquid will spread over a solid surface. Interfacial bonding is due to the adhesion between the reinforcement phase and the matrix. For adhesion to occur during the manufacturing of a composite, the reinforcement and the matrix must be brought into an intimate contact. During a stage in composite manufacture, the matrix is often in a condition where it is capable of flowing towards the reinforcement and this behavior approximates to that of the flow of a liquid. A key concept in this contact is wettability. Once the matrix wets the reinforcement particle, and thus the matrix being in intimate contact with the reinforcement, causes the bonding to occur (Hashim et al., 2001; Oh et al., 1987). Different types of bonding will occur and the type of bonding varies from system to system and it entirely depends on the details such as the presence of surface contaminants. The different types of bonding observed are mechanical bonding, electrostatic bonding, chemical bonding, and inter diffusion bonding (Burr et al., 1995). The bonding strength can be measured by conducting the tests like single particle test, bulk specimen test, and micro-indention test (Dusza and Sajgalik, 1995).

Poor wettability of most ceramic particulates with the molten metals is a major barrier to processing of these particulate reinforced MMCs by liquid metallurgy route. The characterization and enhancement of wettability is therefore, of central importance to successful composite processing (Asthana and Rohatgi, 1993). Wettability is shown in the Figure 1 below and it is customarily represented in terms of a contact angle defined from the Young-Dupre equation which is expressed as follows:
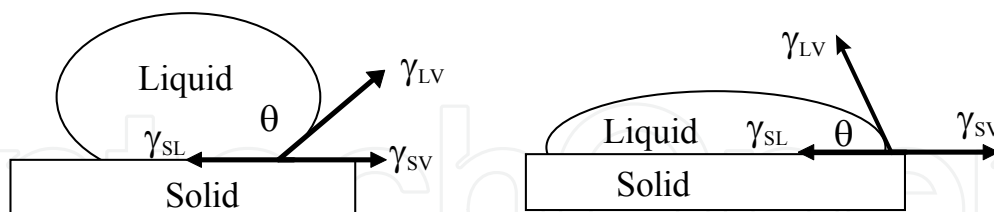

Figure 1. A sessile drop to the left is an example of poor wetting (θ>90°) and the sessile drop to the right is an example of good wetting (θ<90°) (Rajan et al., 1998).

$$\gamma_{lv} \cos \theta = \gamma_{sv} - \gamma_{sl} \qquad (1)$$

Where γ_{SV} = Solid/Vapor surface energy, γ_{SL} = Solid/Liquid surface energy and γ_{LV} = Liquid/Vapor surface energy.

The wetting behavior of a liquid on a solid can be characterized by the wetting or contact angle that is formed between the liquid and the solid substrate. A "sessile drop" is a continuous drop of liquid on a flat, solid surface under steady-state conditions. To neglect the effects of gravity, the gravitational forces should be small compared to the surface tension of the drop. If

this condition is satisfied, the drop will approach a hemispherical shape which represents its smallest area and lowest surface free energy. The sessile drop is placed on the solid substrate and the angle between the solid surface and the tangent to the liquid surface at the contact point is measured. This is known as the contact angle or wetting angle. The contact angle can vary between 0 and 180° and is a measure of the extent of wetting. The conditions of good wetting ($\theta<90°$) and partial wetting ($\theta>90°$) are illustrated in Figure 1. Complete wetting (also referred to as spreading) is obtained at an angle of 0° and complete non-wetting occurs at an angle of 180°. The contact angle is the vector sum of the interfacial surface energies between the solid/liquid (γ_{sl}), liquid/vapor (γ_{lv}), and solid/vapor (γ_{sv}) phases. Young's equation represents a steady-state condition for a solid/liquid interface in stable or metastable thermodynamic equilibrium. Temperature changes have been shown to affect the contact angle of many different systems. The temperature effect, in most cases, can be explained by a reaction at the liquid/solid interface. Thermally activated reactions can occur because many systems are not at chemical equilibrium. The reactions that contribute to wetting (decrease of the contact angle) are those that increase the driving force for wetting (γ_{sv} - γ_{sL}), which is acting at the surface of the liquid drop and the solid substrate. The reactions that contribute to the driving force for wetting are the ones in which the composition of the substrate changes by dissolution of a component of the liquid. On the contrary, if the reaction results in a change of the liquid's composition by dissolution of the solid substrate, but with no change in the composition of the substrate, there is no contribution to the driving force for wetting.

As mentioned above, if the solid substrate is an active participant in the reaction, the free energy of the outer surface of the liquid drop will contribute to the driving force for wetting. As the drop expands on the substrate, the perimeter remains in contact with the unreacted solid and thus the reaction continues to contribute to the driving force for wetting. Examination of phase diagrams representing the interaction between the constituents of the liquid and solid surfaces can help to predict the wetting behavior of a system.

Moreover, measurement of wettability of powders consisting of irregular and polysized particles is extremely difficult. Several techniques have been proposed in the thermodynamic literature to measure wettability. However, these techniques have been applied mostly to non-metallic liquids and their application to metal ceramic systems with reference to pressure casting of composites has been quite limited. The engineering approaches to increasing wettability can be broadly classified into two categories. One method is the surface modification of the reinforcement phase and the other technique is melt treatment. Surface modifications of reinforcements include heat treatment of the particulates to determine surface gas desorbtion, surface oxidation and coating of particles with materials that react with the matrix. Melt treatment is usually done to promote reactivity between the metal and the particulate surface. The wetting reaction must be constrained to prevent reinforcement degradation during the fabrication of subsequent utilization (Ho and Wu, 1998).

2.8. Particulate reinforcement

The improvement in toughness due to the particulate reinforcement depends on the residual stresses surrounding the particles, the weight fraction of the particles, size and

shape of the particles (Suery and Esperance, 1993). Particles can be spherical, disk-shaped, rod shaped, and plate shaped. Each particle forces the crack to go out of plane, and can force the crack to deflect in more than one direction and thus increase the fracture surface energy (Gogopsi, 1994). Plate and rod shaped particles can increase the composite toughness by another mechanism called as 'pullout' and 'bridging'. The residual stress around the particles results from thermal expansion mismatch between the particles and the matrix, which helps to resist the crack propagation. The term 'particulates' is used to distinguish these materials from particle and referred as a large, diverse group of materials that consist of minute particles. The second phase particle can produce small but significant increase in toughness and consequently increases its strength through crack deflection processes. The particles, sometimes given a proprietary coating can be used for improving strength. When compared to whiskers-reinforcement systems, particle reinforcement systems have less processing difficulties and should permit to add higher weight fractions of the reinforcing phase. The orientation of particles appears as flat plates (Matthew and Rawlings, 1999; Pardo et al., 2005).

3. Experimental procedure

3.1. Materials selected for processing composites

Aluminum – 11.8% silicon (LM6)

The main materials used in this project are LM6 aluminum alloy as a matrix material and SiO$_2$-quartz as a particulate reinforced added in different percentages. Pure (99.99%) aluminum has a specific gravity of 2.70 and its density is equals 2685kg/m^3. The details of the LM6 alloy properties and composition is shown in Table 1 and Table 2.

Composition LM6	
Al	85.95
Cu	0.2
Mg	0.1
Si	11.8
Fe	0.5
Mn	0.5
Ni	0.1
Zn	0.1
Lead	0.1
Tin	0.05
Titanium	0.2
Other	0.2

Table 1. Composition of LM6(Sayuti, Sulaiman, Baharudin, et al., 2011)

PHYSICAL PROPERTIES	VALUES
Density (g/cc)	2.66
MECHANICAL PROPERTIES	**VALUES**
Tensile strength, Ultimate (MPa)	290
Tensile Strength, Yield (MPa)	131
Elongation %; break (%)	3.5
Poisson's ratio	0.33
Fatigue Strength (MPa)	130
THERMAL PROPERTIES	**VALUES**
CTE, linear 20°C (μm/m-°C)	20.4
CTE, linear 250°C (μm/m-°C)	22.4
Heat Capacity (J/g- °C)	0.963
Thermal Conductivity (W/m-K)	155
Melting Point (°C)	574

Table 2. Physical, Mechanical and thermal properties of LM6 (Sulaiman, et al., 2008)

Quartz

Pure and fused silica is commonly called quartz. Quartz is a hard mineral which is abundantly available as a natural resource. It has a rhombohedra crystal structure with a hardness of 7 on the Mohs scale and has a low specific gravity ranging from 2.50 to 2.66. It provides excellent hardness when incorporated into the soft lead-alloy, thereby making it better suited for applications where hardness is desirable. It also imparts good corrosion resistance and high chemical stability. It is a mineral having a composition SiO_2, which is the most common among all the materials, and occurs in the combined and uncombined states. It is estimated that 60% of the earth's crust contain SiO_2. Sand, clays, and rocks are largely composed of small quartz crystals. SiO_2 is white in color in the purest form. The properties of pure quartz are listed in the Table 3.

Properties of quartz	
Molecular weight	60.08
Melting Point °C	1713
Boiling Point °C	2230
Density gm/cc	2.32
Thermal Conductivity	0.01 W/cm K (bulk)
Thermal Diffusivity	0.009 cm2/sec (bulk)
Mohs Hardness @ 20 °C	7 Modified Mohs
Si %	46.75
O %	53.25
Crystal Structure	Cubic
Mesh size	230
Size	65 microns (65 μm)

Table 3. Properties of quartz

Preparation of materials

The materials used in this work were Aluminum LM6 alloy as the matrix and SiO$_2$ as reinforcement particulates with different weight percentages. The tensile test specimens were prepared according to ASTM standards B 557 M-94 (ASTM, 1991). Sodium silicate and CO$_2$ gas was used to produce CO$_2$ sand mould for processing composite casting. The aluminum alloy, LM6, was based on British standards that conform to BS 1490-1988 LM6. Alloy of LM6 is actually a eutectic alloy having the lowest melting point that can be seen from the Al-Si phase diagram. The main composition of LM6 is about 85.95% of aluminum and 11.8% of silicon.

The SiO$_2$ particulate used as a second phase reinforcement in the alloy matrix was added on the molten LM6 by different weights fraction such as 5%, 10%, 15%, 20%, 25%, and 30%. The mesh size of Silicon Dioxide particulate is 230 microns and the average particle size equal to 65 microns (65μm).

Fabrication of composites

Only one type of pattern was used in this project and the procedure for making the pattern involves the preparation of drawing, selection of pattern material and surface finishing. Carbon dioxide moulding process was used to prepare the specimens as per the standard moulding procedure. Quartz-particulate reinforced MMCs were fabricated by casting technique. Six different weight fractions of SiO$_2$ particle in the range from 5%, 10%, 15%, 20%, 25%, and 30% by weight were used. In this research work, the particulates were preheated to 200 °C in a heat treatment muffle furnace for 2 hours and it was transferred immediately in the crucible containing liquid LM6 alloy.

3.2. Testing methods

Tensile testing

Tensile test was conducted to determine the mechanical properties of the processed SiO$_2$ particulate reinforced LM6 alloy composites. Test specimens were made in accordance to ASTM standard B557 M-94. A 250 KN servo hydraulic INSTRON 8500 UTM was used to conduct the tensile test. The tensile testing of the samples was performed based on the following specifications and procedures according to the ASTM standards, which of one crosshead speed of 2.00 mm/minute, grip distance 50.0 mm, specimen distance 50.0 mm and temperature 24 ^{0}C.

Hardness measurement

The hardness testing was done on a Rockwell Hardness Tester. The hardness of composites was tested by using MITUTOYO ATK-600 MODEL hardness tester. For each sample, ten hardness readings were taken randomly from the surface of the samples. Hardness values of different types of the processed composites are determined for different weight fraction % of titanium carbide particulate containing aluminum-11.8% silicon alloy and graphs were

plotted between the hardness value and the corresponding type of particulate addition on weight fraction basis.

Impact testing

The impact test was conducted in accordance with ASTM E 23-05 standards at room temperature using izod impact tester. The casting processing steps and testing shows are shown in Figure 2.
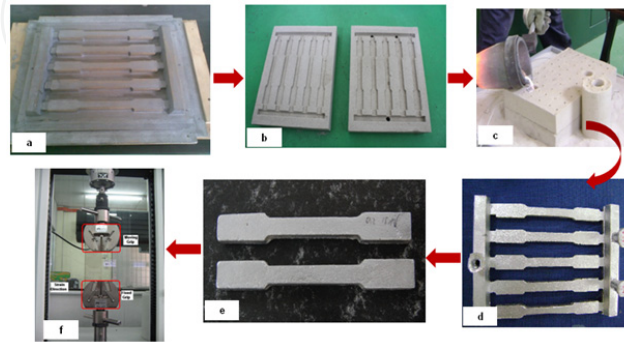

Figure 2. The casting processing steps; (a) Pattern of mould (b) sand mould : drag and copper (c) melting and pouring in the sand mould (d) tensile specimens with gating system (e) tensile specimen after removing of gating systems (f) tensile testing

Density measurement

The density of a material is defined as its mass per unit volume. A&D-GR 200 – Analytical Balance was used to conduct the density measurement. The theoretical density of each set of composites was calculated using the rule of mixtures (Rizkalla and Abdulwahed, 1996). Each pellet was weighed in air (W_a), then suspended in Xylene and weighed again (W). The density of the pellet was calculated according to the formula:

$$Density = \frac{Wa}{(Wa - Ww)} \times density\ of\ Xylene \tag{2}$$

Thermal diffusivity measurement

Thermal diffusivity of composite materials is measured using the photo flash method. The photoflash detection system consists of a light source, sample holder, thermocouple, low noise pre amplifier, oscilloscope, photodiode and a personal computer. The temperature rise at the back surface of the sample is detected by the thermocouple. The detected signal is amplified by a low-noise preamplifier and processed by a digital oscilloscope (Carter and Norton, 2007; Yu et al., 2002).

The voltage supplied to the camera flash is always maintained below 6 Volts before switching on the main power supply. The sample is machined to acquire flat surface to obtain better quality result and it is attached directly to the thermocouple. The camera flash is located at 2 cm in front of the sample holder. Before starting the equipment, the set up

was tested using a standard material such as aluminium. Measurement was carried out every 10 minutes to allow the sample to thermally equilibrate at room temperature. The data was analyzed before running the next measurement.

Photoflash detection system is not an expensive method and the standard thermal diffusivity value for aluminum is equal to 0.83 cm²/sec for thickness greater than 0.366 cm (Muta et al., 2003). In the photo flash system, the excitation source consists of a high intensity camera flash. This method is well suitable for aluminum, aluminum alloys and aluminum-silicon particulate metal matrix composites (Collieu and Powney, 1973). The thermal diffusivity values can be obtained for different thicknesses of the test samples. The thermal diffusivity α determines the speed of propagation of heat waves by conduction during changes of temperature with time. It can be related to α, the thermal conductivity through the following equation (Michot et al., 2008; Taylor, 1980).

The photo flash technique was originally described by Parker and it is one of the most common ways to measure the thermal diffusivity of the solid samples. The computer is programmed to calculate the thermal diffusivity, α, using the equation:

$$\alpha = \frac{\left(1.37 \, x \, L^2\right)}{[(3.14)^2 \, x \, t_{0.5}]} \tag{3}$$

Where L = thickness in mm and $t_{0.5}$ = half rise time in seconds.

Scanning Electron Microscopy (SEM)

LEO 1455 variable pressure scanning electron microscope with Inca 300 Energy Dispersive X-ray (EDX) was used to investigate the morphological features. Results and data obtained from the tensile tested samples were correlated with the reported mechanical properties for each volume fraction of silicon dioxide percentage addition to the LM6 alloy matrix.

4. Results

Tensile properties

The average value of tensile strength (MPa) and Young's Modulus (MPa) versus weight fraction of SiO₂ is shown in the Figures 3 and 4.
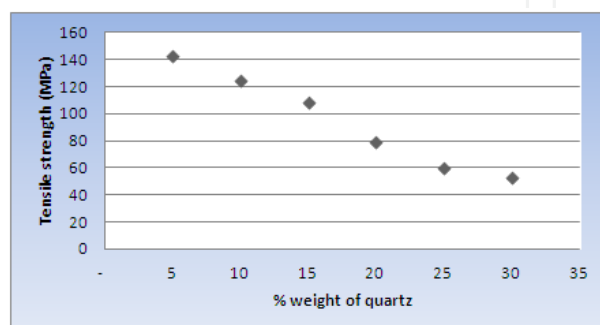

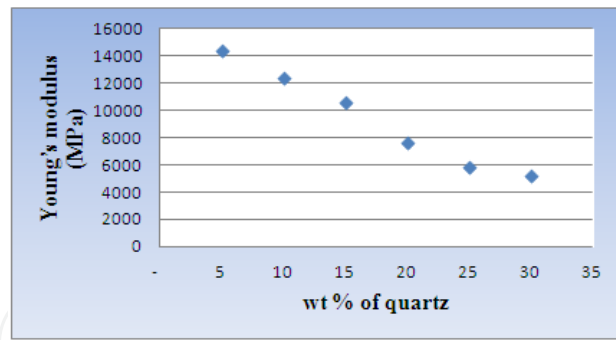**Figure 3.** Tensile strength Vs % weight

Figure 4. Young"s modulus Vs % weight

The graph plotted between the average tensile strength and modulus or elasticity values versus variation in weight fraction of quartz particulate addition to LM6 alloy indicates that both the properties decreases with increase in the addition of the quartz particulate. The increase of closed pores content with increasing quartz particulate content would create more sites for crack initiation and hence lower down the load bearing capacity of the composite. The fluctuation maybe due to the non-uniform distribution of quartz particulates, due to experimental errors and also depends on the cooling rate of the castings (ASTM, 1991; Seah, et al., 2003). When particulates increase, particles are no longer isolated by the ductile aluminum alloy matrix, therefore cracks will be not arrested by the ductile matrix and gaps would propagate easily between the quartz particulates. This residual stress affects the material properties around it and the crack tips and the fracture toughness values would be altered. Consequently, these residual stresses would probably contribute for the brittle nature of the composites. It should be noted that the compressive strength of the quartz particulate dominates which is more than the tensile strength of the LM6 alloy matrix and hence the tensile strength decreases with more amount of addition of quartz particulate afact which is well supported is well supported and evidenced from the literature citation (Rizkalla and Abdulwahed, 1996; Seah, et al., 2003).

Hardness

Similarly, for a given SiO_2 reinforcement content, some differences in the hardness values were observed depending upon the particle size of the constituents. From the Table 4, data on hardness of quartz particulate reinforced composites made in sand mold is listed. It was found that the hardness value increased gradually with the increased addition of quartz particulate by weight fraction percentage as shown in Figure 5.

The maximum hardness value obtained based on the Rockwell superficial 15N-S scale was 67.85 for 30% weight fraction addition. The EDS spectrums for 30% wt of SiO_2 are shown in Figure 5. Their respective elemental analysis is shown in Table 4. It was observed that the grain-refined composite casting has higher weight percentage of Si compared with the original LM6 casting. These results indicate the interrelationship between the thermal properties and hardness.

Impact strength

Impact strength data of quartz particulate reinforced composite castings processed was determined and it is listed in the Table 4. From the plotted graph shown in the Figure 6, it is

found that the impact strength values were gradually increased with the increased addition of quartz particulate in the alloy matrix. The maximum value of impact strength was 24.80 N-m for 30% weight fraction addition of quartz particulate to the alloy matrix. A reason for the increased volume impact–abrasive wear of the SiO2 particle reinforced composites lies in the propensity of the carbides to fracture and spall as a result of the repeated impact from the quartzite. In the monolithic ferrous-based alloys, the matrix can absorb substantial damage in the form of plastic deformation. This plastic deformation is in fact beneficial in that, the matrix will get harder as a result, and wear, fatigue type processes ending as a material removal mechanism. In the SiO_2 particle reinforced composites, however, the high weight fraction of SiO_2 limits the amount of plastic deformation that the matrix can absorb. This leads more quickly to SiO_2 reinforcement fracture, matrix– SiO_2 particle delamination, and S_iO_2 particle spalling. As a consequence, volume impact–abrasive wear increases at a more rapid rate for the composite materials as the hardness increases. However, for the very 'hardest' S_iO_2 particle reinforced composites, impact–abrasion resistance is very good. The summary of mechanical properties of quartz particulate reinforced composite castings processed was determined and it is listed in the Table 4.

	5	10	15	20	25	30
HR value	44.65	49.85	52.73	55.38	60.52	67.85

Figure 5. Hardness Vs wt % of quartz

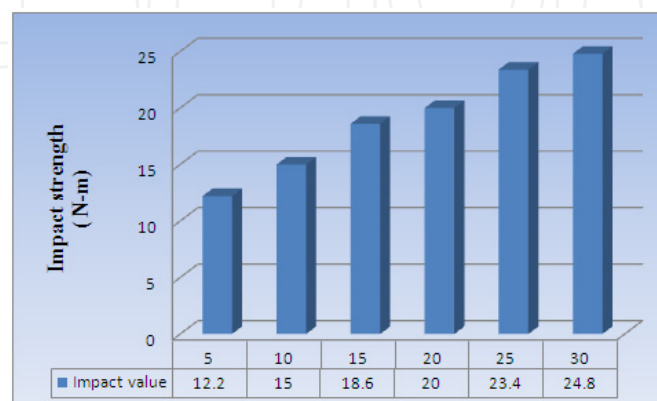|	5	10	15	20	25	30
Impact value	12.2	15	18.6	20	23.4	24.8

Figure 6. Impact strength Vs Weight fraction % of quartz

Wt % of quartz	UTS (MPa)	Yield (MPa)	Young's modulus MPa	Fracture stress MPa	Ductility %	Reduction in area %	Rockwell Hardness	Impact (N-m)
5 %	142.99	132.00	14351	189.50	1.214	2.863	44.65	12.20
10 %	124.74	129.60	12350	164.60	1.412	2.864	49.85	15.00
15 %	108.47	118.50	10635	142.20	1.422	3.042	52.73	18.60
20 %	78.97	109.60	7621	128.40	1.632	3.264	55.38	20.00
25%	59.53	100.50	5853	115.30	1.824	3.625	60.52	23.40
30%	52.64	92.65	5242	104.60	1.741	3.482	67.85	24.80

Table 4. Mechanical properties of quartz particulate composites

Density

Figure 8 gives the influence of quartz addition on the density. The graph shows that as the quartz-silicon dioxide content was gradually increased, the density of the Aluminum composite decreased. Slight decrease was observed in the density because quartz-silicon dioxide has a slight lower density value than LM6 (the density of LM6 is 2.65grs/cc and of quartz is 2.23grs/cc).

The investigation of the aluminum composite was well documented. The percentage of the closed pores in the sintered composites increased with increasing quartz content. This can be attributed to silica being harder than aluminum and non deformation at all under the applied compaction load. The morphological features of quartz particles were significantly different from those of Aluminum and as a result, the interparticle friction effects were different. Therefore, the increase in the amount of closed pores with increasing quartz content would justify the observed decrease in density (Rizkalla and Abdulwahed, 1996).
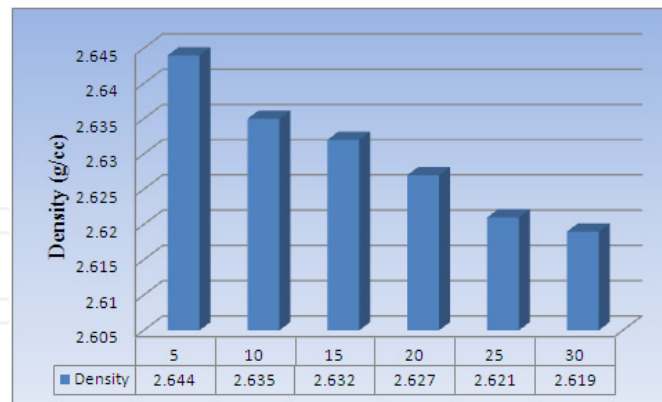

Figure 7. Graph plotted on density versus %wt fraction of SiO_2

Thermal properties

Quartz particulate reinforced composite castings made in grey cast iron mold were tested and analyzed for thermal properties. Graphs are plotted between the weight fraction % addition of quartz and thermal diffusivity and thermal conductivity values. It is found that the thermal diffusivity of the quartz composites decreased with the increased addition in the

alloy matrix. Reversely, the thermal conductivity of the quartz composites decreased with the increased addition of quartz particulate in the alloy matrix. Quartz particulates are a ceramic reinforcement phase and on addition of this in the alloy matrix reduces the thermal conductivity. The data for thermal diffusivity and thermal conductivity of the quartz particulate reinforced composites made in sand mold is given in the Table 4. These are illustrated in the plotted graphs and are shown in Figure 8 and 9. The thermal diffusivity and thermal conductivity for 30% weight fraction addition of quartz are 0.2306 cm²/sec and 52.9543 W/mK respectively and it is well supported from the literature citation (Collieu and Powney, 1973). The summary of physical properties of quartz particulate reinforced composite castings processed was determined and it is listed in the Table 5.
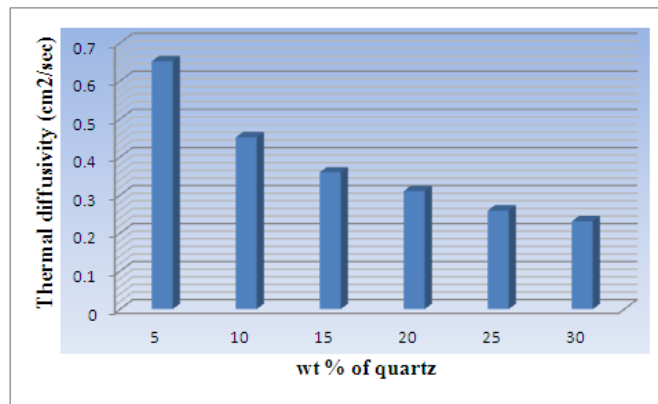
Figure 8. Thermal diffusivity Vs Wt Fraction % of quartz
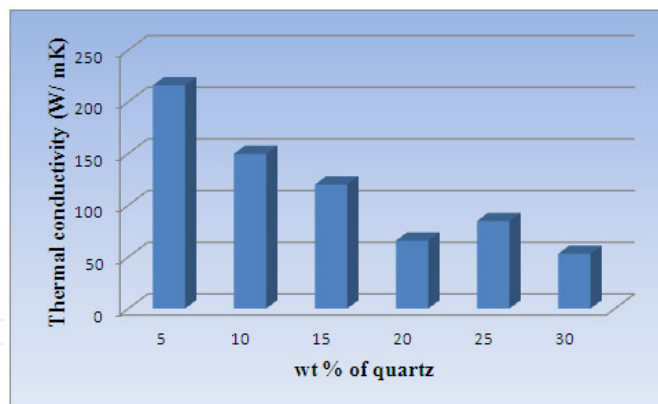

Figure 9. Thermal conductivity Vs Wt Fraction % of quartz

Wt % of quartz	Density (g/cc)	Thermal diffusivity cm²/sec	Thermal conductivity (W/ mK)
5 %	2.644	0.6513	215.826
10 %	2.635	0.4514	149.584
15 %	2.632	0.3595	119.933
20 %	2.627	0.3102	65.6860
25%	2.621	0.2590	84.6830
30%	2.619	0.2306	52.9543

Table 5. Physical properties of quartz particulate composites

Scanning electron microscopy (SEM)

Scanning Electron Microscopy and energy dispersive spectroscopy was employed to obtain some qualitative evidences on the particle distribution in the matrix and bonding quality between the particulate and the matrix. Besides this the fracture surface of the composite was analyzed by using SEM to show the detail of chemically reacted interfaces. Thus, in order to increase the potential application of MMCs, it is necessary to concentrate on the major aspects, like particle size of quartz and quartz distribution concentration.

The fracture surfaces or fractographs are shown in the Figures 10-15 after tensile testing the specimens having different weight fraction of quartz particulate. It was observed that the increase of SiO_2 content would create more sites for crack initiation and would lower the load bearing capacity of MMCs. In addition the number of contacts between quartz particles would increase and more particles were no longer isolated by the ductile aluminum alloy matrix. Therefore, cracks were not arrested by the ductile matrix and they would propagate easily between quartz particulates. Decrease of SiO_2 content to less than 30% in the matrix and a particle size of 230 micron could increase the tensile strength. Hence cracking on the surface is not too dominant. This phenomenon is shown in Figure 10. The problem on interfacial bonding between the particulate quartz and the matrix during the solidification of composites can be ignored because the phenomena of cracking occurs only in a small part of the surface (Seah, et al., 2003). In contrast, when the content of quartz was increased (30%), interfacial bonding concept would be an important phenomenon because the surface cracking will be distributed on the surface of the parts. The other problem caused by the interaction between Aluminum alloy and quartz particle is not a significant one and it is removed while solidification during the pouring process and due to slip inter bonding/ inter granular movement which is illustrated with the aid of Figure 11.
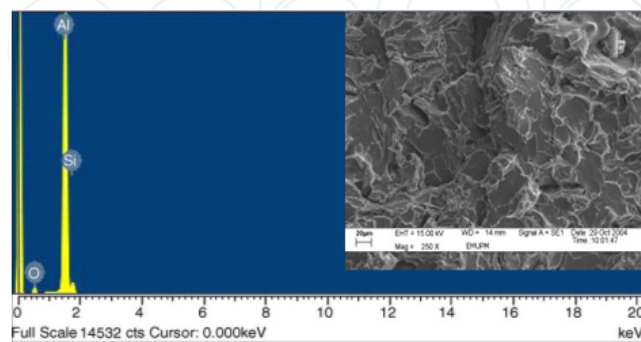

Figure 10. EDX Spectrum and Fractograph of 5wt% quartz particulate reinforced in quartz -LM6 alloy matrix composite at 250X magnification by SEM after tensile testing.
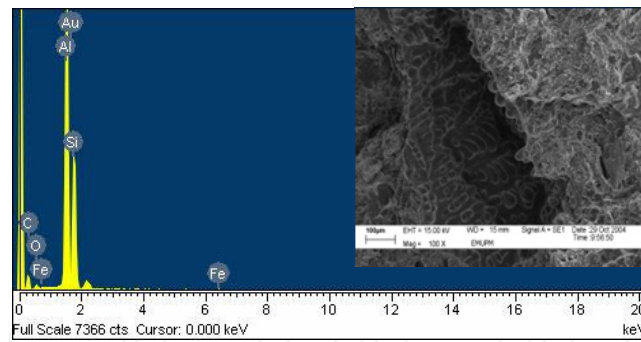
Figure 11. EDX Spectrum and Fractograph of 10wt% quartz particulate reinforced in quartz -LM6 alloy matrix composite at 100X magnification by SEM after tensile testing.
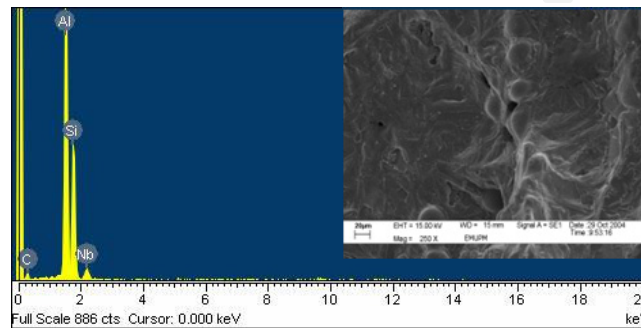

Figure 12. EDX Spectrum and Fractograph of 15wt% quartz particulate reinforced in quartz -LM6 alloy matrix composite at 250X magnification by SEM after tensile testing.
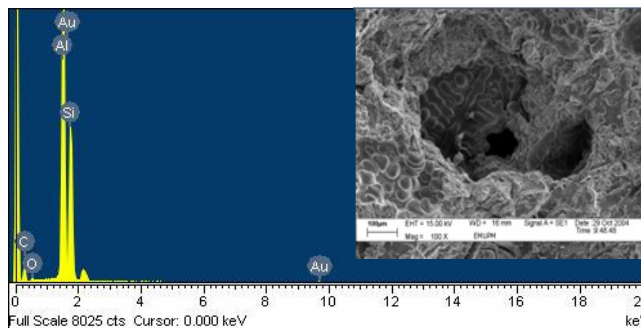

Figure 13. EDX Spectrum and Fractograph of 20wt% quartz particulate reinforced in quartz -LM6 alloy matrix composite at 100X magnification by SEM after tensile testing
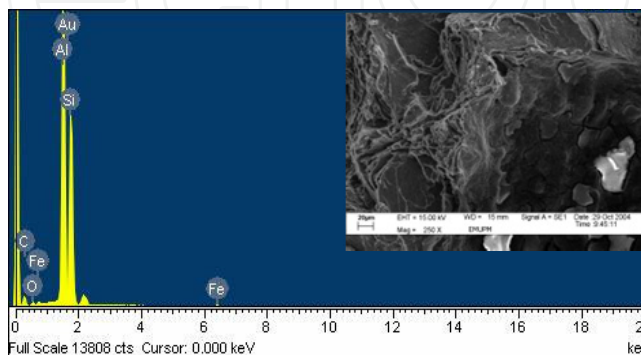

Figure 14. EDX Spectrum and Fractograph of 25wt% quartz particulate reinforced in quartz -LM6 alloy matrix composite at 250X magnification by SEM after tensile testing
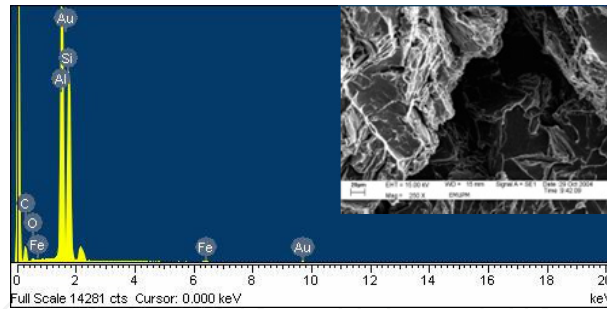
Figure 15. EDX Spectrum and Fractograph of 30wt% quartz articulate reinforced in quartz -LM6 alloy matrix composite at 250X magnification by SEM after tensile testing

5. Conclusions

In this study, the compressive strength of the silicon dioxide particulate reinforcement dominates and influences more effectively than the tensile strength of the LM6 alloy matrix phase. Hence the values of tensile strength and modulus of elasticity are decreased with the increased addition of SiO_2 particulate from 5 to 30% by volume fraction basis. This fact from the present experimental research is well supported and validated from the literature. The mechanical behaviour of the processed composite had a strong dependence on the volume fraction addition of the second phase reinforcement particulate on the alloy matrix. On the other hand, decreasing the SiO_2 particulate content less than 30% by weight along with the particle size constraint as 230 mesh-65 microns would increase the tensile strength and cracking on the surface might not be too dominant. The hardness value of the silicon reinforced aluminum silicon alloy matrix composite is increased with the addition of quartz particulate in the matrix.

The density of these composites decreased slightly with increasing quartz content. Slight decrease was observed in the density because quartz-silicon dioxide has a slightly lower density value than LM6. For a given particle size combination, the thermal diffusivity and thermal conductivity decreases as SiO_2 wt % of the composite increases. The particle size ratio of the constituents becomes an important factor for thermal properties, especially above 10wt. % SiO_2. A higher Al/ SiO_2 particle size ratio results in segregation of SiO_2 particles along the LM6 boundaries. This yields lower thermal conductivity with respect to the homogeneously distributed reinforcement. Therefore, a thermal conductivity value that is less than the expected one might be attributed to the micro-porosity in the segregated structure. Similar tendencies were also observed for the results of hardness tests.

In future, it is strongly recommended that tensile tests be performed by reinforcing the second phase quartz particulate addition to the LM6 alloy matrix by limiting it up to 15 wt%. In addition, compressive strengths testing of the processed composite samples can be done to highlight the benefits, advantages and applications of these composites. It is also worthwhile to conduct heat treatment studies of these processed composites and this will be in the scope of future research work.

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