CHAPTER II

LITERATURE REVIEW

2.1 HISTORY AND APPLICATIONS OF ALUMINIUM AND ITS ALLOYS
About less than 14 metals have densities ≤ 4.5 gm/cc Table 2.1. Out of these, aluminium, magnesium and titanium are commonly used materials. Beryllium is harmful and inconvenient to handle as such used in limited amounts for heat shields and structural components of rockets. Yttrium is a scarce material and has properties attractive to the nuclear powered aircraft project. But the majority of these metals are inappropriate for structural purpose because they have low melting points or chemically reactive.

Table 2.1: The Light Metals

<table>
<thead>
<tr>
<th>Metal</th>
<th>Density (gm/cc)</th>
<th>T_m(°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Titanium</td>
<td>4.50</td>
<td>1667.0</td>
</tr>
<tr>
<td>Yttrium</td>
<td>4.47</td>
<td>1510.0</td>
</tr>
<tr>
<td>Barium</td>
<td>3.50</td>
<td>729.0</td>
</tr>
<tr>
<td>Scandium</td>
<td>2.99</td>
<td>1538.0</td>
</tr>
<tr>
<td>Aluminium</td>
<td>2.70</td>
<td>660.0</td>
</tr>
<tr>
<td>Strontium</td>
<td>2.60</td>
<td>770.0</td>
</tr>
<tr>
<td>Cesium</td>
<td>1.87</td>
<td>28.5</td>
</tr>
<tr>
<td>Beryllium</td>
<td>1.85</td>
<td>1287.0</td>
</tr>
<tr>
<td>Magnesium</td>
<td>1.74</td>
<td>649.0</td>
</tr>
<tr>
<td>Calcium</td>
<td>1.54</td>
<td>839.0</td>
</tr>
<tr>
<td>Rubidium</td>
<td>1.53</td>
<td>39.0</td>
</tr>
<tr>
<td>Sodium</td>
<td>0.97</td>
<td>98.0</td>
</tr>
<tr>
<td>Potassium</td>
<td>0.86</td>
<td>63.0</td>
</tr>
<tr>
<td>Lithium</td>
<td>0.53</td>
<td>181.0</td>
</tr>
</tbody>
</table>
For more than sixty years, aluminium ranks at second to mild steel in the market sector. The rapid demand for aluminium strengthened because of its distinctive set of properties which makes it few of the most adaptable of engineering and construction material. Alloying elements like zinc, magnesium, copper and silicon can radically alter the properties of pure aluminum. Aluminium is light weight with 2.7gm/cc as its specific gravity, which is only one third to iron. It is lightest of all common metals except magnesium. Aluminum is an engineering material of prime significance by virtue of its exceptional properties like good strength, good thermal and electrical conductivities. As there is no allotropic phase transformations in aluminium control of microstructure and properties rely on precipitation reactions. The material derives its strength from work hardening, age hardening and solid solution strengthening.

2.2 ALUMINUM AND METALLURGICAL BACKGROUND

The international system of designation for aluminium wrought alloys is given below in Table 2.2. Each wrought alloy is specified by a four digit number code, with an added letter and number indicating the temper of the alloy.

Table 2.2: Designation of Wrought Aluminium Alloys

<table>
<thead>
<tr>
<th>AA Designation</th>
<th>Major Alloying Elements</th>
</tr>
</thead>
<tbody>
<tr>
<td>1xxx</td>
<td>None</td>
</tr>
<tr>
<td>2xxx</td>
<td>Cu</td>
</tr>
<tr>
<td>3xxx</td>
<td>Mn</td>
</tr>
<tr>
<td>4xxx</td>
<td>Si</td>
</tr>
<tr>
<td>5xxx</td>
<td>Mg</td>
</tr>
<tr>
<td>6xxx</td>
<td>Mg + Si</td>
</tr>
<tr>
<td>7xxx</td>
<td>Zn</td>
</tr>
<tr>
<td>8xxx</td>
<td>Other</td>
</tr>
</tbody>
</table>

In this four digit classification scheme, the major alloying element(s) is represented by the first digit. The remaining three digits are used as serial numbers to identify particular alloy types. Out of the above series of materials 2xxx, 6xxx, 7xxx, 8xxx comprise alloys that are heat treatable.

Silicon and magnesium are the primary alloying elements of 6000 series alloys. They are most commonly used for extruded components due to good formability, medium
strength, good surface finish, and good corrosion resistance [9]. Aluminium 6061 is one of the most widely used heat treatable alloy with good toughness characteristics, and commonly used for medium to high strength requirements. Automobile components to machinery and equipments sports products and consumable durables are most common applications.

2.2.1 Major Alloying Elements and Their Effects
Aluminium is hardly used for engineering applications without being alloyed with other metals. Silicon (Si) copper (Cu) and magnesium (Mg) are the major alloying elements in aluminium alloys.

Silicon (Si):
Silicon imparts good castability and is the most essential single alloying element used in majority of aluminium casting alloys. The Al-Si alloy systems are classified into three major categories: Hypoeutectic, Eutectic, Hypereutectic basing on the Si content in weight percentage.

Copper (Cu):
Copper has prominent effect compared to other alloying elements on properties of aluminum alloys. Strength and hardness of both heat and non heat treated aluminium castings are influenced by Copper content, at ambient and elevated operating temperature. Copper when alloyed with aluminium reduces the corrosion resistance of the alloy.

Magnesium (Mg):
Magnesium imparts significant strengthening and improves work-hardening properties of aluminium. Magnesium enhances weldability and can impart good corrosion resistance [10][11]. During the hardening phase Silicon when combines with magnesium forms magnesium silicide (Mg$_2$Si), which provides the required strengthening.
Other alloying elements include zinc, nickel, tin, titanium, boron etc. Titanium and boron are used as grain refiners. Titanium and boron when used in combination provide better grain refining.

2.3 METAL MATRIX COMPOSITES
2.3.1 Introduction

Composites mostly refers to material structure made out from distinct constituents, reinforcements are dispersed in a continuous phase called matrix, and unique features are acquired from the properties of its constituents, morphology of reinforcements and properties of the matrix and reinforcement interfaces [12][13]. Typically, composite consists of a matrix and reinforcing materials intimately bonded together. Depending on the matrix materials used, there are three types of composite materials namely, polymer, ceramic and metal matrix.

With novel technologies and development of various processing techniques, composites with improved properties have appeared as attractive contenders to materials engineers. To fabricate better performance MMCs and to address the shortcomings of fiber reinforced MMCs, particulate reinforced metal matrix composites (PRMMCs) have replaced as viable alternatives [14][15]. The benefits of particulate reinforced MMCs over continuous fiber reinforced composites includes low cost of reinforcing particulates, simple and low cost production and processing methods, and isotropic properties [16][19][17].

Most of the studies as of late were concentrated on the development of aluminium matrix composites [18]. Magnesium based composites attracted less Interest mainly due to its intrinsic properties of low deformability at room temperature and low corrosion resistance although they can offer similar or higher property improvement when compared to aluminum based composites [19]. Particulate strengthened MMCs are usually produced by Powder metallurgy and conventional casting techniques [19].
2.3.2 Particulate Strengthened Metal Matrix Composites
Particulate strengthened MMCs are synthesized by combining an alloy with fixed quantity of ceramic particles in powder form to enhance the properties. This result is a significant increase in modulus and wear resistance. Compared to fiber reinforced composites the improvements achieved are less yet they are cost effective, and have isotropic properties and can be processed by most metal working processes like machining, welding and deformation. The primary impetus for research and development of particulate strengthened MMCs is to produce a material with tailorable properties without the drawbacks of fiber reinforced MMCs.

2.4 METAL MATRIX TYPES
A variety of metals particularly low density metals or alloys are typically chosen as the matrix material for the composite. Properties of some commonly used metallic matrices are as follows [20] [21] [22].

Aluminium Alloys
Aluminium alloys are mainly used in automobile and aero space sector because of density, good strength, and toughness and corrosion resistance. To improve the strength of Aluminium and its alloys, ceramic particles are added to form a composite material. 6xxx series aluminium alloys have become attractive candidates in automotive and aerospace structural applications due to good forming and joining properties and also due to excellent corrosion protection. Aluminium 6061 is a common alloy of this series that is utilized where excellent weldability and good corrosion properties are required.

Titanium Alloys
Titanium based composites have found wide application as materials for high temperature structures. Alloys of titanium are mostly used in the aerospace components due to low density, strength at elevated temperature and exceptional oxidative and corrosive resistance. However, the material is expensive.
Magnesium Alloys
Magnesium is the lightest among various non ferrous metals, commonly used in gearbox housings for aerospace applications, chain saw housings and electronics equipment.

Copper Alloys
Copper is a good electrical and thermal conductor. It can be easily cast and formed. Copper based composite materials having good thermal conductivity and wear resistance are used in electronics as electrical contacts and elements of electronics system.

2.5 REINFORCEMENTS
Reinforcements are primarily available in particulates form, whiskers and fibers. Sometimes high melting point metals in the form of fine particles are also used as reinforcements [23].

Particulate reinforced MMC’s are being utilized for a range of industrial applications. The particulate reinforcements are mostly oxides, carbides or borides. Reinforcements such as Al₂O₃, SiO₂, ZrO₂, SiC, B₁C, TiC, WC, TiB₂, BN, etc have been investigated [24]. During processing, chemical reactions can occur between the matrix and the particulate. For instance SiC is reactive with Aluminium alloys, where as Al₂O₃ is more stable in the same case. For structural applications, reinforcement volume percent of 10-30% and particle size in the range of 10-30µm is used. MMCs with higher volume fractions are also available commercially, for example electronics packaging applications, where higher particle volume percentages are used, such as around 70% by volume.

2.6 METHODS FOR FABRICATION OF MMCs
Composite properties are not only influenced by the quantity and type of reinforcing material but also by their fabrication method. Different fabrication methods are available for making particle-reinforced MMCs [19] [25] [26], the details of most of these fabrication routes are proprietary. Essentially, there are two basic methods of
processing depending on the state of the matrix namely, solid state and liquid state methods. A typical example of solid state processing route is the powder metallurgy while melt stirring also known as stir casting is most commonly used liquid state processing route. In addition to these, there is a dual phase process where the matrix is in maintained between solidus and liquidus and is called compocasting. The choice of fabrication method is governed by particle size, particle shape, particle volume, particle-matrix reactivity and commercial viability of the chosen route. Some of the processing methods are described below.

2.6.1 Liquid Metallurgy
Most of the commercially available composites are processed by casting methods because of the advantages compared to solid state methods. Firstly, liquid metal is easy to handle and less expensive compared to solid metal powders. Secondly products can be cast in desired shapes. Due to inadequacy of control over processing parameters unwanted chemical reaction at metal reinforcement interface results in lack of reproducibility. Due to these reasons, liquid state processing methods are limited to low melting point alloys.

Liquid state processes can be divided into four categories on the basis of physical combination of the matrix and the reinforcement [27]. These are:

1. Stir casting (vortex method)
2. Infiltration
3. Spraying
4. In-situ

2.6.1.1 Stir Casting
In this process, particulate reinforcements are slowly introduced into the molten metal while the melt is being stirred. Generally molten metal do not wet reinforcement particles, to incorporate particles into the melt and improve wetting mechanical force is used and it is generally accomplished by stirring. Presently stir casting process is an inexpensive manufacturing method for MMCs.
Vortex method is simplest process which involves intense agitation of the liquid metal and then adding reinforcement particles into the vortex. Schuster and Skibo [28] [29] patented a process in 1988 in which Silicon carbide particles are consolidated into liquid aluminum in vacuum using a specifically designed impeller. This process has resulted in reducing inclusions such as impurities, oxides and gases in the melt due to vacuum and by minimizing the vortex. The successful implementation of this process depends on controlling unwanted developments such as gas porosity due to gas entrapment during mixing, inclusions, clustering of particle and interfacial reactions between molten metal and reinforcements due to long contact times.

Reinforcement blending can also be performed with the temperature of the melt being maintained in semi solid state. The advantage of the process is that, particle movement either settling or floating in semi solid melt is prevented by the solid phase. In this process particle incorporation is important as the particles cannot be easily incorporated in the liquid metal due to difficulty in breaking the oxide layer and in substituting the particle gas interface with the particle liquid interface. The equation for force balance in vertical direction on a partially immersed particle in the melt as stated by Ilegbusi and Szekely [30] is given by Equation 2.1.

\[ \sum F = F_B + F_G + F_H + F_S + F_E \]  \hspace{1cm} (2.1)

Where
- \( F_B \) = Buoyancy force on the immersed part
- \( F_G \) = Gravity on the whole particle
- \( F_H \) = Hydrostatic pressure of the liquid above the contact area
- \( F_S \) = Vertical component of the surface tension
- \( F_E \) = Drag force caused by the motion of the liquid

Other phenomena governing melt stirring process are fluid flow and particle migration. Unreinforced metals have viscosity similar to water which is about 0.01 poise and they can be considered as Newtonian fluids. Addition of ceramic particles to these metallic melts increases their viscosity due to interaction between the particles and with the matrix creating higher resistance to shear stress.
In semi solid temperature range, composite slurry is simply a mixture of a liquid and solid phase and its behaviour is explained in the same way as unreinforced melts. When shear is applied, there is a change in the morphology of the solid phase as a result of dendrite fragmentation, ripening and abrasion and collision and coalescence of solid particles. As quoted by reference [14], Mada and Ajersch advanced an analytical model of thixotropic behavior in semi solid composite slurries. They concluded that in semi solid slurries the particle addition has little impact on thixotropic behavior and the major factor is the primary solid phase. In another study by Moon et al. which is also quoted by reference [6] it is observed that viscosity of slurries containing SiC particles combined with an aluminum alloy in the semisolid temperature range was lower than that of unreinforced slurry with the same volume % of solid. This can be explained by the location of ceramic particles between dendrite arms preferentially limiting the contact and agglomeration of dendritic solid particles.

The last phenomena governing melt stirring method is the particle migration. After mixing, the composite might be at rest before solidification. Depending on the density the reinforcement might either settle to the bottom or float on the top. In order to produce homogeneously reinforced composites, an understanding of the interaction of the growing solid metal and the particles is essential. It is observed experimentally that there is critical velocity, \( V_c \) at the interface above which particles get engulfed and below which they are pushed. There are some models governing this phenomenon but they are not easy to compare with the experimental results as the parameters do not vary independently and castings do not solidify at steady state as assumed by these models.

2.6.1.2 Liquid Infiltration

Liquid infiltration processes are related to a porous reinforcing phase (generally fibrous) which is placed in the mold and molten metal is forced through the mould to fill the voids and form a composite. There are two types of infiltration processes: pressure less and pressure assisted infiltration. In pressure less infiltration, the liquid matrix infiltrates a porous preform of reinforcement without the aid of external force. In pressure assisted infiltration, external force is applied by mechanical means or by
gas. Composites produced by pressure assisted infiltration are denser compared to pressure less infiltration.

Squeeze casting is a type of pressure infiltration process that involves squeezing the molten metal into particulate/fibrous preform [13]. The major advantage of this process is shorter processing cycle which results in high-volume production of components. Other advantages include capability to manufacture relatively complicated shapes, reduced shrinkage or residual porosity, and negligible interfacial reactions between matrix and reinforcement.

2.6.1.3 Spraying

Springer [31] originally developed the spray forming process for conventional metals and alloys subsequently put to commercial use by Osprey Metals [32] which is being put to use for manufacturing MMCs.

In spray forming process schematically shown in Figure 2.1, metal droplets along with reinforcement are sprayed on a substrate where they solidify to form a composite. The advantages of this forming methods are: (i) fine grained matrix microstructures with low segregation, (ii) contact time between reinforcement and molten metal is less (iii) steps are less compared to powder metallurgy process (iv) near net shapes are possible (v) low oxide contents and (vi) the wide choice of suitable matrix alloys. (vii) The absence of organic binders. The shortcomings include difficulty in obtaining homogeneous distribution of reinforcement and high porosity. Cost of production by spraying is reported to be between sintering and casting [19].
2.6.1.4 In-Situ Processes

These processes are characterized by the in-situ formation of the reinforcement phase. The XD™ (exothermic dispersion) is a process where exothermic reaction between two constituents produces a third constituent. The XD™ process was developed by ‘Martin Marietta Corporation’ [33][34] to manufacture in-situ composites. This approach to manufacture MMCs is rather different than the conventional methods mentioned in the previous sections. In this process, the metallic matrix is mixed with compounds with which it reacts exothermally. Temperature of the mixture when raised above the melting point of the matrix or to a point where a self-propagating reaction takes place, the constituent components react exothermally to form a dispersion of submicroscopic reinforcing particles in the matrix hence the name "XD". Since the particles of the reinforcing phase are formed exothermally at high temperatures, they are very stable during subsequent processing and use at elevated temperatures. XD™ process can be used to form broad range of reinforcing compounds [34]. However, TiB₂ and TiC are the two that have received wide attention and are formed according to the reactions given below

\[ Ti + 2B + Al \rightarrow TiB_2 + Al \]  \hspace{1cm} (2.2)

and

\[ Ti + C + Al \rightarrow TiC + Al \]  \hspace{1cm} (2.3)
There is ambiguity and not much of information is available in the literature regarding (i) the cost of products produced by the XD™ process, (ii) porosity levels in the as-reacted materials, and (iii) the control of spacing and the size of reinforcing particles. The production of a variety of MMCs by this process has been reported. These include matrices of Al, Ti, Fe, Cu, Pb, and Ni and intermetallics such as TiAl, Ti₃Al, and NiAl [35].

A different way to produce in-situ composites is the reaction between liquid metal and gas. An example of this process is Lanxide’s Dimox (Directed Metal Oxidation) process in which composite is produced by direct oxidation of metal. The process typically involves adding Mg to high temperature aluminum melt to make the alumina skin unstable by the formation of a porous compound thus helping the metal to move into an array of ceramic particles by capillary action. A major advantage of this process is there is homogeneous dispersion and spacing of reinforcement particles which can be adjusted by the rate of reaction or solidification.

2.6.2 Solid State Fabrication
Solid state processing includes a series of steps leading to final product. These processes utilize blended elemental powders for the fabrication of particulate reinforced MMC’s.

2.6.2.1 Powder Metallurgy
Attempts to produce particle reinforced MMCs by blending particulate reinforcements into liquid metal had not been promising as the common metals and alloys do not wet non metallic particles. Powder metallurgy route developed helped to some extent in overcoming this difficulty. According to Lloyd [19] the advantages of this method are (i) higher volume fraction of reinforcements can be added, resulting in high elastic modulus and reduced thermal expansion coefficient (ii) most of the alloy can be used as matrix (iii) reduced interfacial reactions.

In solid state process, matrix and reinforcements in powders form are blended to get a homogeneous mixture. To obtain homogeneous blend with minimum agglomerates,
the size of metal and ceramic particles should be chosen carefully. The ratio of appropriate sizes depends on the blending process. Lewandowski et al. [115] avers SiC/Al composites particles having 0.7:1.0 ratio gives better distribution of reinforcement compared to 0.3:1.0 ratios. Generally, the size of atomized metal powder is available in the range of 20-40 µm and the reinforcing particles size vary between 3-20 µm with aspect ratios < 5:1. Usually the blending operation precedes Cold isostatic pressing, canning, degassing, and consolidation at high temperature, finally the product is extruded. An extrusion ratio 20:1 or higher gives better results [19]. A high extrusion ratio tends to break any of reinforcement particle clusters, results in a good bond between the constituents by disrupting the oxide film between the particles and promotes reinforcement distribution.

One of the main disadvantages of powder metallurgy is that the fabrication route is relatively complex. Other obstacles include limited availability, high cost of metal powders and high processing costs resulting in relatively expensive products compared to products prepared via conventional casting routes [25].

2.6.2.2 Diffusion Bonding
Diffusion bonding (foil-fiber-foil) route is mainly used to fabricate monofilament reinforced metal matrix composites. Lately diffusion bonding via the foil-fiber-foil method is being used to fabricate AA6061-boron fiber composites. It is a complex process and to realize homogeneous fiber distribution and high fiber volume fraction is difficult to achieve. It is difficult to create complex shapes and components by this process [12].

2.6.3 Secondary Processing
Secondary processing of composites lead to disintegration of reinforcement clusters, reduced porosity, and enhanced bonding among particles. It is observed that rolling particulate reinforced MMCs in temperature range of 0.5Tm and low rolling speeds give better results [36].
2.7. GRAIN REFINEMENT AND HALL-PETCH EFFECT

Cast metal matrix composites have a coarse grain structure and are dependent on the rate of cooling. To improve the strength grain refining is carried out. The effect of grain refinement on strength of a material is generally illustrated by the Hall Petch relationship. Grain refinement can substantially improve the properties such as hardness and strength of metals. In contrast to cold working which trades strength with ductility grain refinement usually increases the strength as well as ductility especially in super plastic deformation. Hall [37] and Petch [38] first discovered the strengthening in polycrystalline metals with small grain size and introduced the famous Hall-Petch relation as follow:

$$\sigma = \sigma_0 + KD^{-1/2}$$  \hspace{1cm} (2.4)

Where
\[\sigma = \text{flow stress of polycrystalline material}\]
\[K = \text{constant and}\]
\[D = \text{grain size}.\]

It is observed from Equation 2.4 reduction in grain size increases strength and this has led to increased enthusiasm for producing materials with smaller grain size. The grain size can be modified by subjecting them to predefined thermo-mechanical processing.

The basis of the hardening effect in Hall-Petch relation was elucidated well by Li and Chou [39] in one of their review. According to them surrounding grain boundaries and dislocation pile-ups adjacent to grain boundaries block gliding dislocation propagating between grains. With the increasing numbers of dislocation pile ups at the grain boundaries, the concentration of stress increases and when a critical stress is reached another dislocation is activated in the neighboring grain and glides until it meets another grain boundary. As the size of grains decrease, the total grain boundary area gets increased substantially and more obstructions are encountered by the dislocations. Higher flow stress is thus required for the dislocations to move through the grain boundaries.
Grain refinement in polycrystalline materials can be achieved by many methods. One common practice of grain refinement of polycrystalline material is by adding nucleating agents (grain refiners) to the base metal before or during casting process. It is observed that these additives serve as heterogeneous nucleating sites during the cooling which promotes the fine grain structure or as obstacles which hinder the grain growth. Superheating is also used to refine the grain structures of polycrystalline magnesium alloys [40].

Another common grain refinement method first introduced by Valiev et al. [41] is severe plastic deformation (SPD). SPD processes can produce fine grained materials and sometimes even ultra fine grained structures. Ultra fine grained (UFG) materials usually exhibit improvement in properties like strength and ductility.

2.8 SEVERE PLASTIC DEFORMATION (SPD) OF METALS
SPD Processed materials have been the focal point for the research community for the last ten years due to the distinctive properties attainable by SPD processing.

Severe plastic deformation (SPD) of materials leading to Ultra fine grained (UFG) structure is of interest in the field of materials engineering [42][44][43][44]. The importance is due to distinctive properties in strength, ductility and even super plastic behavior. ECAP is one of the most preferred SPD process, it involves pressing the material through an ECAP die consisting two intersecting channels intersecting at a certain angle but of equal cross sections, typically the angle is 90°. The ECAP process allows us to induce very large plastic deformations to a work-piece without altering the overall geometry of the work piece.

One of the most important advantages of ECAP is that plastic strain of very high magnitude can be induced into the materials without any significant modification in geometry of the sample. Very large strains with ultrafine grain structure can be obtained by ECAP and hence advantageous over conventional metal working processes.
Formal definition of SPD can be as follows: “Any metal forming method under considerable hydrostatic pressure that may be used to impose a very high strain on a bulk solid without any significant change in the overall dimensions of the sample and having the ability to produce exceptional grain refinement” [45].

### 2.8.1 Equal Channel Angular Pressing (ECAP)

Equal channel angular pressing (ECAP) is an encouraging method for producing fine grained materials. It was first pioneered by Segal in the year 1981 [49]. In contrast to rolling, extrusion drawing the main purpose of ECAP is to accumulate deformation in material without any reduction in work piece cross-section. This process involves forcing a sample through an ECAP die Figure 2.2 with two cross-sections intersecting at angle (φ) ranging between 90° and 120°. Some dies have a rounded corner having an angle (ψ) at outer radius.

The work piece is introduced into the channel at the top and is forced through the channel below by a punch. During pressing the billet is subjected to plastic strain by simple shear but retains its geometry. Repetitive pressings can be carried out, each pass refining the grain. During shear deformation by this process, the grain size gets reduced to sub micron levels resulting in improved mechanical properties which can be controlled by proper selection of processing parameters.

The strain imposed by an ECAP die on a sample during a single pass primarily depends on the intersecting angle φ between the two channels. Besides, the outer radius represented by angle ψ has major implications on effective strain, homogeneity and texture.

The accumulated strain after N passes can be obtained by using the Equation 2.5 as proposed by Iwahashi [47].

$$\varepsilon_p = \frac{N}{\sqrt{3}} \left[ 2 \cot \left( \frac{\phi}{2} + \frac{\psi}{2} \right) + \psi \csc \left( \frac{\phi}{2} + \frac{\psi}{2} \right) \right]$$  \hspace{1cm} (2.5)
Figure 2.2: Principles of ECAP Showing the Two Critical Angles $\phi$ and $\psi$ [46]

Figure 2.3: Dependence of Equivalent Strain Achieved in a Single ECAP Pass on the Die Angles [48]

Experiments to date using ECAP has mostly used dies having $\phi = 90^\circ$ and $\psi = 0^\circ$. A single pass with this configuration gives an equivalent strain of 1.1 the amount of strain induced per pass reduces with an increase in angle $\phi$ as shown in Figure 2.3 [48].

Segal [49] observed that rotating the sample between successive pressings modifies the shear directions and planes thus giving freedom to achieve distinct microstructure. The microstructural characteristics during ECAP depends on the precise processing conditions, and shear induced in each pass through the die [42][50]. Four processing
routes can be obtained by rotating the piece about its longitudinal axis between two successive passes.

Route A: there is no rotation of the sample between successive pressings
Route B_A: alternate rotations of ± 90°
Route B_C: forward rotations of + 90°
Route C: the billet is rotated by 180° between passes

As shown in Figure 2.4 Furukawa et al. [51] outlined the processing routes that are most commonly considered in terms of the rotation between the pressings. The affect of pressing by these processing routes results in modifying the shear planes and shear directions thus developing different types of microstructures.

![Figure 2.4: The four ECAP Processing Routes [51]](image)

2.9 CHALLENGES IN STIR CASTING
Compocasting is the easiest, economical and most commercially used technique for manufacturing metal matrix composites. However, technical issues are associated with producing a homogeneous composite with high density. Several factors need to be considered while producing MMCs by stir casting process:
1. Effect of wettability between reinforcement and matrix
2. Tendency to form porosity formation in cast composites
3. Possibility of reactions between reinforcement and matrix
4. Difficulty in attaining a homogeneous reinforcement distribution in the matrix
To achieve optimum properties, reinforcement distribution should be uniform in the matrix. Excellent bonding between reinforcement and matrix should be achieved. Interfacial reactions and porosity must be minimized.

2.9.1 Fluidity

A common problem in stir casting processes is the addition of reinforcements to the liquid metal which can affect the fluidity of the melt. This can lead to undesirable inhomogeneous reinforcement distribution and agglomeration.

Few researchers [52][54] studied the fluidity of aluminium alloys and their composites. As the alloy starts solidifying, the drop in flow is due to choking of the flow tip of the stream. The viscosity rises rapidly when solid concentration reaches a critical value at the leading tip, resulting in abrupt stoppage of flow [55][56]. The shape, size and volume of reinforcement particles effect the fluidity of aluminium based composites [57][58].

The fluidity of molten metal is the ability to flow in long channel with thin cross sections [59]. Fluidity of an alloy is influenced by the temperature, the composition, the density and the viscosity of the alloy, thermal properties of the metal, mold and factors such as applied metal head and channel diameter [60]. For a composite, the addition of particles to the melt implies that additional parameters which influence the fluidity above those presented for the pure alloy must also be considered, namely, volume fraction, size and shape of particles, reactions, segregation, clustering, gas and inclusions [58].

The fluidity of metals can be evaluated by two common tests spiral fluidity test and vacuum fluidity test [56][59]. Fluidity of the metal is measured in terms of the length of the channel along which the molten metal flows before it is stopped due to solidification [59]. To evaluate fluidity of pure metal, Flemings developed the following Equation 2.6 assuming that acceleration, friction, and flow separation are negligible [59].
Where:

\( L_f \): Fluidity length (fluidity)
\( \rho_s \): Solid metal density
\( a \): Channel cross section Radius
\( v \): Velocity of metal in liquid state
\( h \): Coefficient of heat transfer
\( C \): Specific heat of metal
\( H \): Latent heat of fusion
\( \Delta T \): Molten metal super heat
\( T_0 \): Mould temperature
\( T_m \): Melting point of metal

### 2.9.2 Fluidity of Composite

Addition of solid particles into the melt affects the fluidity by the modification of flow properties and solidification time because the ceramic particles added to the liquid phase do not have the same thermal properties as the liquid metal. Thus, the solidification time of a composite melt will be different from that of the pure alloy due to change in latent heat induced by the reinforcement volume fraction there is change in superheat and thermal conductivity of the melt. For describing composite fluidity, Yarandi et al. [54] proposed Equation 2.7 by introducing the reinforcement volume fraction and their properties in Equation 2.6.

\[
L_f = \frac{\rho_s \, a \, v}{2h(T_m - T_0)} (H + \Delta T) 
\]

(2.6)

\[
L_f = \frac{(\rho_m \phi_m + \rho_d \phi_d) a v}{2h(T_m - T_0)} [H_m (1 - \phi_d) + (C_m W_m + C_d W_d) \Delta T] 
\]

(2.7)

Where:

\( L_f \): Fluidity length (Fluidity)
\( \rho \): Density of solid metal
\( \phi \): Volume fraction
\( C_m \): Specific heat of matrix
Cd : Specific heat of Particles
H : Latent heat
Wm: Matrix weight fraction
Wd: Weight fraction of reinforcement particles

From the above equation it can be deduced that density, reinforcement content and thermal properties of reinforcements effect composite fluidity. Rohatgi and Asthana [58] reported that the experimental observations are consistent with the values predicted by the above equation, provided velocity (v) and coefficient of heat transfer (h) do not change significantly. Further Rohatgi and Asthana [58] suggest that the important factors not considered in Equation 2.7 which affect composite fluidity are agglomeration, settling of reinforcement particles, existence of gas, oxides and changes induced due to reactions.

2.9.3 Influencing Factors of Reinforcement on Fluidity

2.9.3.1 Particle Volume Fraction
The fluidity of particulate composites at a given temperature is less compared to their unreinforced alloy [54][61][62].

Figure 2.5: Fluidity vs. Vol. % of SiC Particles in A356 and A357 Cast in Permanent Mold [63]
Carity [63] observed that an increase in SiC volume fraction fluidity of A356 and A357 alloys decreased when cast in permanent molds as shown in Figure 2.5.

2.9.3.2 Particle Surface Area
For a given reinforcement percentage, decrease in particle size or an increase in angularity decreased composite fluidity [52] [53] [64]. Surappa and Rohatgi [53] observed a linear decreased in fluidity with an increase in surface area of particles when cast in permanent molds (Figure 2.6).

![Figure 2.6: Variation of Spiral Fluidity (cast in permanent mold) as a Function of Specific Surface Area of Ceramic Particles [53]](image)

2.9.3.3 Particle Size
According to Surappa and Rohatgi [65] for given quantity of reinforcement fluidity of aluminium-copper-mica composites when cast in metal molds decreased with decrease in the reinforcement size (Figure 2.7). Yarandi et al. [66] observed that for A356 alloy-SiC particulate composites containing 15.0 vol. % SiC particles of 9μm diameter showed lower flowability compared to that of composite containing 21.0 vol. % silicon carbide reinforcement of 15μm diameter, demonstrating that particle size strongly influences the fluidity of the composite melt.
For a given volume fraction decrease in reinforcement size results in increase of total particulate surface area causing higher resistance to flow because of stagnant boundary layer around particles.

2.9.3.4 Particle Shape
A change in surface area to volume due to morphology of reinforcing particles influences the fluidity of composite melt.
Increase in angularity of reinforcing particles decreases the fluidity for a given volume fraction of particles and temperature Figure 2.8 [65].

### 2.10 WETTING PHENOMENA OF CERAMIC PARTICLES BY METALS

Processing composites using conventional casting process is an attractive and economical as it offers a wide choice in materials selection and processing conditions and it is relatively inexpensive. Non wetting nature of the ceramic reinforcements is the primary obstacle to the liquid-metal techniques. Improved wetting between reinforcement particles and the matrix is crucial for a good bond during fabrication of composites [67].

Despite wettability being an important factor during composite processing, not many significant studies have been conducted, and quite a few basic questions still remain unexplained. Properties of composites are controlled to a larger degree by interface characteristics between matrix and reinforcement [68][69][70]. From metallurgical perspective the interfaces in composites depend on several factors [71]. Wetting as well as spreading are well defined thermodynamic processes, but their use is diverse and inconsistent [27].

Wettability is described as the extent of interfacial contact between the solid and liquid. The angle of contact as shown in Figure 2.9 of a liquid drop at equilibrium resting on a solid surface is determined by Equation 2.8 generally referred to as Young-Dupre equation [72].

\[
\gamma_{sv} = \gamma_{sl} + \gamma_{lv} \cos \theta \quad (2.8)
\]

- \( \gamma_{sv} \) = Solid vapor surface tension / specific energy at the solid-vapor interface
- \( \gamma_{sl} \) = Solid liquid surface tension / specific energy at the liquid-solid interface
- \( \gamma_{lv} \) = Liquid vapor surface tension / specific energy at liquid-vapour interface
- \( \theta \) = Contact angle
According to Young [72] work of adhesion, $W_{AD} / W_A$ is the force of bonding between liquid and solid phase. The work of adhesion is the quantity of work necessary to separate a unit area of the solid liquid interface into two surfaces [73].

$$W_{AD} = \gamma_{sv} + \gamma_{lw} - \gamma_{sl}$$  \hspace{1cm} (2.9)

Combining Equation 2.8 and 2.9 gives

$$W_{AD} = \gamma_{lw} (1 + \cos \theta)$$  \hspace{1cm} (2.10)

Equation 2.10 expresses the force of bonding between solid and liquid phases in terms of liquid surface tension and contact angle. The contact angle $\theta$ indicates wettability as shown in the Figure 2.9.

- $\theta = 0^0$ is perfect wetting
- $\theta = 180^0$ represents no wetting
- $0^0 < \theta < 180^0$ shows partial wetting

![Figure 2.9: Sessile Drop Tests for Measuring Contact Angle][59]

It can be observed that lower contact angle results in good wettability. According to Dellanay et al. [75] in vacuum wetting is effected by two factors namely strength of liquid solid interaction at the interface and surface tension of the liquid.
Wetting properties are commonly measured by sessile drop experiments they involve placing a liquid metal drop on a solid surface and measure the angle. Normally sessile Drop procedure is used in temperature range of 400-2000°C [74][77]. Figure 2.10 shows wetting and non-wetting systems.

### 2.10.1 Factors Retarding Wettability

Normally the metal oxides present on the surface of melt or adsorbed gases on ceramic surface results in reduced wetting of reinforcement particles by metals. The oxide layer acts as an obstacle preventing the penetration of ceramic particles into molten matrix. Aluminium with high affinity towards oxygen formation of oxides in aluminum alloys is difficult to avoid. For example 50nm thick oxide layer is formed on aluminum alloy in four hours when held at 400°C [78]. Better wetting can be ensured by reducing oxide layer on the melt surface [74].

Furthermore, presence of gas can form a barrier as the particles volume fraction reaches a critical proportion resulting in total rejection of particles from the melt [79]. Consequently it is critical that the gas layers present should be completely dried before composite processing. Zhao et al. [81] proposed that the presence of gas around particles is the main cause for poor wettability. Therefore to achieve good wettability it might be necessary to rupture the gas layers surrounding the particles.
When the gas layers collapse the metal and reinforcement particles are in direct contact resulting in good wettability.

Characteristics of the particle surface also influence wettability. The silicon oxide layer present on the solid surface prevents the wetting of silicon carbide particles by liquid metals. As a result, a certain threshold temperature is required to transit from non-wetting to wetting condition [82]. Wetting is commonly not observed at temperatures below 910°C, for Al-SiC when the melt temperature is raised to 1100°C there is a significant decrease in contact angle from 150° to 34° [84]. Eustathopoulos et al. [83] also demonstrated that the existence of aluminium oxide layer on the melt surface prevents direct contact between aluminium and carbon during synthesis.

2.10.2 Methods to Promote Wettability
Certain methods were proposed to improve wetting of reinforcement with molten metal or alloy [82][94][95][88] which includes

i. Inclusion of alloying elements into the molten alloy
ii. Particle coating
iii. Particle heating

2.10.2.1 Addition of Alloying Elements
Reactive elements when added to the molten metal matrix induce wettability which results in exceptional bonding between the liquid matrix and reinforcement during liquid metallurgy route [93]. For instance, the addition of alloying elements like magnesium, titanium, zirconium etc., to liquid metal reduces surface tension of the melt thus improving wetting.

Magnesium when added to aluminium based composites results in improved consolidation of reinforcement particles when compared to other elements [88][89] titanium, zirconium and lanthanum [81] lead, zinc, copper and bismuth, [90]. It is observed that adding magnesium to aluminium melt promoted wetting of alumina
particles [85][91] and certainly it is observed that magnesium is compatible with aluminium and most of the reinforcements [92][94].

Addition of 3.0 % magnesium to aluminium melt held at 720°C reduced the surface tension [96]. Initial addition of 1wt % magnesium results in very sharp decrease in surface tension. For instance, with 1wt % magnesium the surface tension of aluminium the alloy dropped from 0.860 Nm$^{-1}$ to 0.650 Nm$^{-1}$ [74]. Sukumaran et al. [95] in their work conclude that it is essential to add magnesium while processing A356-SiC particulate composites by stir casting, and observed that 1wt% magnesium is the optimum value for best reinforcement distribution and improved mechanical properties. They also observed that addition of magnesium below certain optimum value resulted in agglomeration and uneven dispersion of reinforcement in the melt.

Levi et al. [92] avers that the formation of magnesium aluminate spinel layer between reinforcement and aluminium melt improves the bonding in Al-Mg alloy based composites. A356 alloy matrix composite containing 80-100µm silicon carbide particles prepared by liquid metallurgy technique demonstrated that adding magnesium helped in reducing surface gas layers present on silicon carbide particles [94].

It is reasoned that addition of magnesium to liquid aluminium alloy during composite synthesis not only scavenges surface oxygen from the particles but also strengthens the matrix. However, excess addition of magnesium to aluminium melt deteriorates mechanical properties by forming low-melting constituents. According to Sukumaran et al. [95] adding 3.0 % magnesium to A356 alloy results in formation of low melting point Mg$_5$Al$_8$ phase. Additionally, Korolkov [94] cautioned that the Casting fluidity of molten aluminium reduces with the addition of magnesium.

A clean surface is necessary for better interaction of particle and melt, thus enhancing wetting. Some of the techniques used to clean the particle surface are heating in suitable atmosphere, etching techniques and ultrasonic techniques, [74]. Formation of silica layer on SiC particles prevents the formation of Al$_4$C$_3$, and also improve wetting between SiC and aluminium due to the reaction between aluminium and SiO$_2$ [77].
2.10.2.2 Particle Coating
Practically, liquid metal do not wet the surfaces of ceramic particles. Wetting can be accomplished by coating ceramic particles with metal. It is a fact that liquid metals quite often wet solid metals and wettability between them depends on mutual solubility [75]. Metals like nickel and copper are most commonly used as metal coating for reinforcement particles typically used in aluminium matrix based composites [103][104][105][106]. Some researchers have proposed silver, copper and chromium coatings on reinforcing particles [107]. Some of the methods used for coatings ceramic particles includes electroplating, chemical vapour deposition, plasma spraying [99] and by sol gel process [108].

2.10.2.3 Particle Heating
Pre heating of particles before incorporating into the melt helps in desorption of adsorbed gasses from particle surface which helps in their transfer. Agarwala and Dixit [90] ascertained the relevance of pre-heating graphite particles before adding to aluminium alloy melt. They observed that the particles were retained in the melt when preheated compared to non-retention of graphite particles in the melt when not preheated. According to Ribes et al. [98] heating of silicon carbide particles modifies the surface composition by the forming an oxide layer when heated to 900°C thus helping in reducing surface impurities. Few investigators [99] [100] suggested that oxide layer on particle has ability to promote wetting between SiC particles and liquid metal. It is also observed that preheating alumina particles improved wetting of alumina particles by aluminium magnesium melt [101][102].

2.10.2.4 Other Methods to Promote Wettability
Mechanical force is applied in order to improve wettability by reducing surface tension. However, Zhao et al. [81] in their experimental work found that the matrix alloy when mixed in fully liquid state by mechanical stirring could not solve poor Wettability. It was observed that wetting improved between SiC particles and aluminium alloys containing Si and Mg when stirring was carried out in semi solid state.
An important variable for processing metal matrix composites by liquid metallurgy route is the mixing time, which is not sufficiently addressed. Many metal ceramic systems encourage interfacial reactions in composite manufacturing to make them wettable. Since the interfacial reactions progresses with time, the contact angle $\theta$ also varies with time, the particles may appear to be non-wetting if the processing time is less, but as time increases Wettability is improved. For example, Al-SiC composite melt had a contact angle of 125° at holding temperature of 800°C and holding time of 125 minutes and after holding time of 160 minutes the contact angle dropped to 55°. Similarly, contact angle for coated particles will be time dependant. During processing if the particle coating has the tendency to dissolve in melt the processing time must be controlled to prevent the coating from complete dissolution [80].

2.10.3 Wetting of Ceramic Particulates in the Production of MMC’s

Good wetting is necessary between reinforcement and liquid metal to form an adequate bond during the fabrication of MMCs. Particle size plays an important role in wetting, as quoted by Mortensen [109] an increase in particle size, decreases the centrifugal force required to immerse the particles. It is indicated that it is possible to correlate wetting data with processing parameters and these indicate that finer particles are harder to incorporate which is consistent with this fact. Mortensen [110] also indicates that the sessile drop experiment only replicates poorly the wetting conditions in metal matrix composites fabrication. He adds that even if $\theta$ is measured, its correlation with processing parameters is not easy to explain. In the case of stirring particulate reinforcements in molten metal, the correlation between particles size, stirring speed, wetting angle and engulfment kinetics of particles is not as simple as shown [30].

On the basis of data available on wetting angle, wetting between reinforcement and molten metal is found to be promoted by either reactivity of the metal with the reinforcement or by the reduction of tenacity in the oxide layer of metal which has propensity to oxidize as in the case of aluminium. Depending on these observations, much work has been done to find means of enhancing wetting of ceramics by molten metal. Most of this work has been classified into two categories: Alloying additions and reinforcement coating.
Effective alloying additions fall into two categories:
1. Additions that assist matrix and reinforcement reactions
2. Additions that modify the oxide layer present on surface of the metal but do not promote reactions with the matrix but

Ilegbusi and Szekely[30] investigated adhesion of metals, especially aluminum on Al₂O₃. They proposed a model for the interface between aluminum and Al₂O₃ and based on this model they attempt to calculate work of adhesion theoretically.

2.11 POROSITY
Porosity is one of the biggest problems faced during aluminium castings production. Casting defects like porosity and oxide inclusions reduce the strength of materials including composites. Largely the properties of cast MMC’s are determined by the distribution of porosity its volume fraction and size [112].

To produce sound castings with good properties it is important to reduce porosity to a minimum. During the casting process Porosity cannot be fully avoided, and for this reason the properties of castings are commonly associated with volume fraction of porosity [128]. However, level of porosity can be reduced by controlling the main sources of this porosity.

In general there are three sources from which porosity arises
i. Gas entrapment while stirring
ii. Evolution hydrogen gas
iii. Solidification Shrinkage

According to Ghosh and Ray [111][113] stirrer size, speed, its position in the slurry and holding time influenced formation of porosity. Their experimental results showed that increase in holding temperature resulted in decrease in porosity level. They recommend the use of turbine stirrer which should be so positioned that 65% liquid should be above and 35% liquid below. According to Lloyd [19] and Samuel et al [114] unsatisfactory casting practices results in various defects including porosity. As
indicated by Pai and Rohatgi [107] the extent of porosity in cast composites depends predominantly on the quantity of reinforcements rather than on the amount of dissolved hydrogen. This is because cast composites have a higher volume of non metallic solids compared to most standard aluminium castings and in cast composites porosity increases linearly with volume fraction of reinforcement.

According to Ray [117][118] the occurrence of porosity in cast composites is associated with the quantity of hydrogen dissolved in the melt and gas being sucked due to improper stirring. Vortex generated in melt due to vigorous stirring tends to trap and draw the gas into the melt.

Several approaches proposed to minimize porosity are
i. Compocasting in vacuum
ii. Large scale bubbling of inert gas into the melt
iii. Solidification of castings under pressure
iv. Extrusion / rolling of cast composites to close the pores

2.12 STRENGTHENING MECHANISMS IN PARTICULATE REINFORCED MMCs
Two approaches for predicting the mechanical properties of materials are continuum approach and micro mechanics approach. These mechanisms are discussed for particulate reinforced metals by Miller and Humphreys [124]. The first mechanism is quench strengthening. A wide difference in CTE exists between metal matrix and particulate reinforcements resulting in the generation of dislocations during solidification. Factors effecting dislocation density are reinforcement volume fraction and reinforcement size. The strength can be given by the Equation 2.11 below:

In the second approach mechanism is grain strengthening is achieved with the addition of grain refiners. Finer grains might improve the strength of the material as formulated by the Hall-Petch equation stated earlier.
\[ \sigma_d = \alpha \, G \, b \, \rho^{1/2} \]  \hspace{1cm} (2.11)

where
\[ \rho = 12 \, \Delta T \, \Delta C \, F_{v} \, / \, b \, d \]
\[ \sigma_d = \text{Strength (0.2\% offset yield strength)} \]
\[ \alpha = \text{A constant between 0.5 and 1} \]
\[ G = \text{Shear modulus of Materials} \]
\[ b = \text{burger’s vector} \]
\[ \rho = \text{Dislocation density} \]
\[ d = \text{Reinforcement size} \]
\[ F_{v} = \text{Reinforcement volume fraction} \]
\[ \Delta C = \text{Product of the thermal mismatch} \]
\[ \Delta T = \text{Temperature change} \]

There are other mechanisms which are effective in certain situations in the case of particle reinforced metals such as Orowan strengthening, Sub structure strengthening and work hardening. For example, Orowan strengthening occurs when the size of the particle is less than 1\(\mu\)m and others occur during secondary processing routes such as heat treatment or work hardening.

The degree of strengthening in composites is closely related to particle distribution and shape. Particle distribution can be specified by volume fraction of particles, average diameter of particles and mean inter particle spacing [119]. The above factors are interrelated and cannot be changed without affecting the others.

For given reinforcement volume fraction smaller particle size gives increased strength. Experiment and theory justifies this effect. An explanation of this can be that the strength distribution of a ceramic particulate population qualitatively follows Weibull’s weakest link effect. A statistical theory of brittle fracture assumes that if there is no interaction between cracks in a rigid body, the composite strength is determined by the element with the longest crack. Weibull statistical distribution is appropriate in representing this situation [120] relating the fracture strength to the number of cracks. If this is adapted to particulate reinforced composites, large
particles are more likely to have critically sized flaws thus severely compromising the strength of composite.

Experimental evidence indicates that voids preferentially nucleate at the sharp corners of ceramic reinforcements and these voids may lead to premature composite failure. According to this, use of spherical reinforcement particles reduces stress concentration thus changing the stress strain distribution all through the composite. In an investigation by Song et al. [121] the difference in strength, ductility and fracture behavior of aluminium 6061 matrix Al_2O_3 reinforced composites reinforced with angular and spherical reinforcements is considered.

![Figure 2.11: True Stress Strain Curve of Spherical and Angular Particle Reinforced Composites [121]](image)

Figure 2.11 shows true stress versus true strain plots for composites reinforced with angular and spherical shaped reinforcements, it is apparent that composites containing angular particles exhibit high yield strength and lower elongation to failure, hence lower ductility. This is attributed to the observation of fracture surface by SEM that unlike angular reinforcements where composite fracture is due to particle fracture, failure due to spherical reinforcements occurs due to nucleation and growth of void in the matrix.

Mummery et al. [122] in their Work indicated that when there is high volume fraction particle clusters in the matrix, these clusters introduce a large constraint on matrix deformation. Thus, void nucleation in clusters can occur at lower strains than elsewhere in the matrix. Ranjit and Surappa [123] fabricated Al–Li–SiC particulate
composites by a modified conventional stir casting process and also observed the clusters of SiC particles are responsible for reduced strength of SiC composites.

Summarizing these, for certain reinforcement volume fraction strength of composite with angularly shaped particles is higher than those where the particles are spherical but the ductility is lower than the case where reinforcement is spherical.

2.13 ENGINEERING PROPERTIES OF PARTICLE REINFORCED MMCs
A brief discussion of some engineering properties of particle-reinforced MMCs is presented here to show how these properties are influenced by the inclusion of ceramic particles into aluminum alloys. Most attractive features of MMCs are the reduction of density, enhancement of specific stiffness, specific strength, Wear and creep resistance, and thermal expansivity. Stiffness is a critical design parameter for many engineering components because the avoidance of excessive elastic deflection in service is the principal overriding consideration. A typical potential application of improved creep resistance is in the development of high-temperature components, such as turbine engine parts where the aim is to replace some heavy components with components made of much lighter materials.

2.13.1 Stiffness
Elastic modulus is one mechanical property that is significantly increased by the addition of ceramic particles into a metallic melts. The enhancement of stiffness achieved by the addition of the reinforcement is retained at high temperatures and this is of great benefit in the design of rotating parts, and structural bodywork. Tables 2.3 and 2.4 [36] list the properties of a few commercially available unreinforced alloys and some particle-reinforced MMCs, respectively. As given in Table 2.4 the modulus of composites increased as reinforcement volume fraction increased which can be calculated using rule of mixtures (ROM) expression. It should be noted that the ROM is appropriate for estimating the Young’s modulus of continuous reinforcement, but it overestimates that of discontinuous reinforcement. Therefore, that is why this has been modified in the Halpin-Tsai equation given below [125].
\[ E_c = \frac{E_m(1-2sqV_p)}{1-qV_p} \]  

(2.12)

Where

\[ q = \frac{(E_p/E_m - 1)}{(E_p/E_m + 2s)} \]

Where moduli of elasticity of matrix, particle and composite are given by \( E_m \), \( E_p \), \( E_c \) respectively, \( s \) is aspect ratio of particles and \( V_p \) volume fraction of particles. To calculate modulus of elasticity Eshelby equivalent inclusion method can also be used [126] and this approach has good reconciliation with experimental data.

### 2.13.2 Elongation

It can be seen from a comparison of Tables 2.3 and 2.4 that a significant limitation of particle-reinforced MMCs is relatively low ductility (as quantified by percent elongation). The tensile elongation decreases with increasing particle content.

**Table 2.3: Properties of Some Unreinforced Alloys [36]**

* 0.2% offset yield strength

<table>
<thead>
<tr>
<th>Alloy</th>
<th>YS* (MPa)</th>
<th>UTS (MPa)</th>
<th>Elongation (%)</th>
<th>E (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6061 (T6)</td>
<td>275</td>
<td>310</td>
<td>20</td>
<td>69</td>
</tr>
<tr>
<td>2014 (T6)</td>
<td>476</td>
<td>524</td>
<td>13</td>
<td>73</td>
</tr>
<tr>
<td>2124 (T6)</td>
<td>325</td>
<td>470</td>
<td>12</td>
<td>72</td>
</tr>
<tr>
<td>2618 (T6)</td>
<td>370</td>
<td>470</td>
<td>9</td>
<td>74</td>
</tr>
<tr>
<td>7075 (T6)</td>
<td>505</td>
<td>570</td>
<td>10</td>
<td>72</td>
</tr>
<tr>
<td>8090 (T6)</td>
<td>415</td>
<td>485</td>
<td>7</td>
<td>80</td>
</tr>
<tr>
<td>A356 (T6)</td>
<td>205</td>
<td>280</td>
<td>6</td>
<td>76</td>
</tr>
<tr>
<td>A380 (F)</td>
<td>160</td>
<td>320</td>
<td>3.5</td>
<td>72</td>
</tr>
<tr>
<td>AZ91</td>
<td>168</td>
<td>311</td>
<td>2.1</td>
<td>49</td>
</tr>
<tr>
<td>AZ61</td>
<td>157</td>
<td>198</td>
<td>3.0</td>
<td>38</td>
</tr>
</tbody>
</table>

It is observed that in heat treatable alloys tensile elongation decreases with an increase in aging time [127]. The opposite changes in stiffness as well as ductility as
reinforcement content increases reflect the interactions between reinforcement and matrix in composite materials.

Table 2.4: Typical properties of some commercially available MMCs [36]

<table>
<thead>
<tr>
<th>Composite</th>
<th>YS (Mpa)</th>
<th>UTS (Mpa)</th>
<th>Elongation (%)</th>
<th>E (Gpa)</th>
<th>Supplier</th>
</tr>
</thead>
<tbody>
<tr>
<td>6061/Al₂O₃/10p (T6)</td>
<td>296</td>
<td>338</td>
<td>7.5</td>
<td>81</td>
<td>Duralcan, Alcan</td>
</tr>
<tr>
<td>6061/Al₂O₃/15p (T6)</td>
<td>317</td>
<td>359</td>
<td>5.4</td>
<td>87</td>
<td>Duralcan, Alcan</td>
</tr>
<tr>
<td>6061/Al₂O₃/20p (T6)</td>
<td>359</td>
<td>379</td>
<td>2.1</td>
<td>98</td>
<td>Duralcan, Alcan</td>
</tr>
<tr>
<td>6061/Al₂O₃/20p (T6)</td>
<td>305</td>
<td>330</td>
<td>3.4</td>
<td>85</td>
<td>Comral85, Comalco</td>
</tr>
<tr>
<td>6061/SiC/15p (T6)</td>
<td>342</td>
<td>364</td>
<td>3.2</td>
<td>91</td>
<td>Cospray, Alcan</td>
</tr>
<tr>
<td>2014/Al₂O₃/10p (T6)</td>
<td>483</td>
<td>517</td>
<td>3.3</td>
<td>84</td>
<td>Duralcan, Alcan</td>
</tr>
<tr>
<td>2014/Al₂O₃/15p (T6)</td>
<td>476</td>
<td>503</td>
<td>2.3</td>
<td>92</td>
<td>Duralcan, Alcan</td>
</tr>
<tr>
<td>2014/Al₂O₃/20p (T6)</td>
<td>483</td>
<td>503</td>
<td>1.0</td>
<td>101</td>
<td>Duralcan, Alcan</td>
</tr>
<tr>
<td>2014/SiC/15p (T6)</td>
<td>466</td>
<td>493</td>
<td>2.0</td>
<td>100</td>
<td>Cospray, Alcan</td>
</tr>
</tbody>
</table>

Previous works have indicated that the composites failure can attributed to cracking of particles, void formation and particle clusters [36][129][115]. Lloyd [36] has suggested that particle fracture is more frequent with coarse particles than with finer ones due to the higher probability of finding crack-initiating defects in the former than in the latter particles.

The failure associated with particle clusters is due to higher stresses generated in such clusters. It has been reported that deformation of matrix between particles which are
closely spaced will be highly constrained resulting in local stress level which is many times more than the flow stresses of matrix [93] which has been proved by continuum modeling [131][132]. It has been observed that for Al-SiCp composites particle cracking is an important failure mechanism for composites containing ≥ 20µm size particles [36].

The geometry of the reinforcement particles in MMCs has been shown to considerably alter the behavior of matrix deformation [130][133][134]. The fact is that in response to external loads the matrix stress and strain fields vary markedly with the geometry of reinforcements [132][135] this in turn alters the fracture behavior particularly near matrix reinforcement interface. As stated earlier Song et al. [121] used SEM and TEM to study the effect of reinforcement morphology on fracture and ductility of particle reinforced aluminium 6061 composite. It was found that the composites containing spherical shaped particles showed significantly higher ductility but lower yield strength and work hardening than composites containing angular reinforcements.

Experimental evidence in the literature shows that generally voids nucleate near sharp corners of the reinforcements [129][132][136][137]. Fisher and Gurland [138] have discussed the factors that tend to favour the formation of voids. Voids often cause premature failure of the composite. Recent FEM modeling has suggested higher ductility in composites with spherical reinforcements due to the lower matrix triaxiality [121][139]. Therefore, a viable way to enhance composite ductility is by incorporating spherical reinforcement particles to reduce stress concentration and thereby redistributing stress all through the composite material. The stress distribution around reinforcing particles in a deforming matrix has been studied by some researchers [140] [141]. The stress concentration at sharp corners of reinforcement particles gives rise to intense localized plastic flow [142][143].

2.13.3 Strength
Strength of engineering material is of prime importance while designing components. A comparison between Tables 2.3 and 2.4 shows the enhancement of yield Strength
due to addition of reinforcement particles is quite substantial. Comprehensive study first carried out by Mc Danels [144] on strength of several discontinuous particles reinforced metal matrix composites reported up to 65% increase in both yield and tensile strength. Subsequent work by different authors essentially confirmed these findings, but the reported experimental data showed a wide scatter due to differences in the material quality, processing routes, and testing parameters.

Christman et al. [142] discussed extensively and modeled mathematically numerous strengthening mechanisms and their behavior that operates on particle reinforced MMCs. The strengthening process in the composite has been modeled based on two different approaches, namely, the continuum approach and the micromechanics approach. Cox [147] originally developed the continuum shear lag model and modified later by Piggot [148] and Nardone [149], gives the composite strength ($\sigma_c$) for a particulate composite as:

$$\sigma_c = \sigma_m \left[ V_p \left(\frac{a+4}{4}\right) + V_m \right]$$

Where $\sigma_m$ is yield stress of matrix, $V_m$ and $V_p$ are volume fraction of matrix and particle volume fraction, and $a$ is aspect ratio of particle. Commonly for particle reinforced MMCs the aspect ratio is in the range of 1.5:1.

The main attraction of using the MMCs stems from the fact that they offer greater strength, especially at room temperature. Presence of reinforcement particles enhances the modulus at higher temperatures but do not improve strength at higher temperatures. The reason is the strengthening mechanisms operating on MMCs get relaxed at higher temperatures. Thus the strength of composite at high temperature is primarily controlled by matrix strength.

2.14. WEAR
Advanced engineering materials having light weight and low cost are the requirements due to current developments in fields of science and technology. They
should be cost effective in various engineering applications where high strength to weight ratio along with good wear properties are required [150][151][152].

Major restriction in the use of aluminium alloys had been their poor wear resistance. Aluminium MMCs have a potential for improved wear resistance over the unreinforced alloy. Although different Wear applications require different reinforcement types to achieve optimal Wear rate reduction, there are many situations where Wear rates are reduced by factors of up to ten by the introduction of the reinforcement. This wear resistance makes MMCs very attractive material for bearings, bushes, and brake drums. In some cases, distribution of reinforcement is controlled so as to provide material of high Wear resistance in selected surface areas while other regions are suitably tough, strong, or thermally conducting.

2.14.1 Wear Mechanisms
During sliding, materials behave differently under diverse sliding conditions. In fact, wear mechanisms such as abrasive, adhesive, corrosive, fretting, erosive, and delamination change as the sliding conditions are varied,. Debris produced by each wear mechanism is of particular shape and chemical composition, and leaves a worn surface with distinct morphology [153]. Hence, an experimental study of debris and worn surface can provide important information to know which wear mechanism operated. In operation, more than one wear mechanism may be operating on a typical engineering contact [154]. In most cases, wear is dominated by more than one wear mechanism which makes the post analysis even more complicated [155].

2.15. WEAR BEHAVIOUR OF SPD MATERIALS
Significant changes in the material properties particularly microstructure and grain size takes place during SPD processing. SPD process improves the mechanical properties, fracture behavior and also effect wear behaviour. Wear is a complex property, factors including load, sliding velocity, lubrication, and mechanical properties affect the wear response of the material. When abrasive wear is the main wear mechanism, material hardness is crucial to the wear rate, when the dominant wear is by delamination, formation and growth of the cracks under surface are
important while the chemical activity of surface may be vital when oxidative wear works. Wear behaviour studies in SPD processed materials need a comprehensive understanding on the effect of SPD process on material and its effects on wear behaviour.

2.15.1 Improvement in Wear Resistance by SPD

Few studies have evaluated the benefit of ECAP process on wear resistance of materials [157][158][159][160][161]. The wear performance of ECAP processed Al alloys with different copper content under different sliding conditions were investigated [156]. The work established the improvement in hardness as the ECAP passes and copper content increased. (Figure 2.12 (a)). With increase in ECAP passes and Cu content and decrease in grain size there is remarkable decrease in wear mass loss (Figure 2.12 (b)).

![Figure 2.12: The Effect of ECAP and Copper Content on (a) Microhardness and (b) Wear Mass Loss of Al–Cu alloys [156]](image)

Some researchers [159][160][161] investigated wear behaviour of aluminium bronze alloy subjected to ECAP processing. They observed as the ECAP passes increased coefficient of friction reduced appreciably (Figure 2.13 (a)) and any reduction in friction coefficient enhances the load bearing capability. The specimens showed the lowest wear rate after six ECAP passes (Figure 2.13 (b)). In particular, after six ECAP passes the seizure load increased from 160N to 253N suggesting that the wear resistance of aluminium bronze improved due to ECAP processing. Copper-chromium alloy was subjected to fretting wear and results indicate that the wear properties improved after ECAP processing due to strengthening and grain refinement [162].
Figure 2.13: Effect of ECAP on (a) Friction Coefficient and (b) Wear Rate of Cu-10Al-4Fe Alloy [159]

Semenov et al. [163] studied tribological behaviour of ECAP processed magnesium (AZ 91D) alloy dispersed with SiC particles. It was observed the coefficient of friction reduced considerably after two ECAP passes. However, wear behavior of ECAP processed AZ61 Mg alloy when investigated under dry sliding conditions indicated that the refinement of microstructure after the ECAP process did not have any effect on wear rate [164].

Wear behavior of Ti-Ni alloy processed up to two ECAP passes was investigated by Cheng et al. [165]. The as-received Ti-Ni alloy had an average grain size of 80μm, while that of ECAP processed Ti-Ni alloy had a grain size of 5μm. Wear loss of Ti-Ni alloy after two ECAP passes decreased by 62.7% and 21.4% under loads of 50N and 250N respectively as compared to as received samples. Detailed observation of worn surfaces revealed that wear mechanism of ECAP processed Ti-Ni alloy is micro-ploughing and delamination, whereas for as-received Ti-Ni alloy wear surface revealed adhesion and delamination.

Purcek et al. [166] observed that dry sliding wear characteristics of Zn alloy after ECAP processing showed reduction in both wear and friction coefficient, particularly at higher loads. It was observed that worn surface of ECAP processed material revealed plastic deformation and adhesion suggesting ECAP process can impede certain mechanisms of wear.
2.15.2 Reduced Wear Resistance by SPD

Experimental results of some SPD processed materials indicate adverse effect on wear behavior. Comparative wear study of cast and ECAP processed Al-Si alloy revealed that an increase in weight loss after ECAP processing. It is also observed that wear resistance decreased after six ECAP passes when compared to as cast alloy, particularly with high applied pressures and long sliding distance [167]. It can be presumed that oxide layer formed on the wear surface due to tribochemical reaction which played predominant role in wear resistance of the alloy, and it concealed strengthening affect of the alloy on wear resistance.

Study conducted by Nickel et al. [168] on the effect of ECAP process on wear properties of sintered Al-Cu composites consisting of Al2O3 and SiC particles in different volumes (5 and 15 vol. %) and fine and nano sizes were used as reinforcements. Oscillating friction and scratch tests performed to assess the wear behavior of composites demonstrated that composites subjected to ECAP process were less wear resistant against oscillating friction movement. Whereas composites processed by ECAP exhibited better wear resistance under abrasive load.

Kim et al. [169] explored sliding wear behaviour under dry conditions of Al alloys with ultrafine grains, and low carbon ferrite martensite dual phase steel. Figure 2.14 displays wear rate versus load of UFG dual phase steel and coarse grained counterpart. The UFG steel exhibited high wear rate compared to coarse grained steels because of relatively low strain hardening of the UFG steel which can lead to lower wear resistance of the UFG steel at high loads.

Investigation on wear properties of UFG aluminium alloys and commercially pure aluminium subjected to accumulative roll bonding (ARB) process at different speeds under dry and lubrication conditions [169][170][171][172] revealed the wear resistance of the accumulative roll bonding processed samples was less compared to non processed samples. Lower wear resistance of UFG aluminium alloy can be attributed to low strain hardening capability of the alloy. Different studies on accumulative roll bonding processed aluminium alloys demonstrated the increase in hardness did not increase wear resistance [164][171][172].
There is difficult in precisely modeling wear mechanisms of SPD materials because of complex wear behaviour. Only one report by Talachi et al. [171] attempted to explain why ARB processed alloy has low wear resistance compared non ARB processed alloy.

### 2.15.3 No effect on Wear by SPD

Some studies also showed that SPD processing had no effect on wear resistance. Sato et al. [173] processed Al-titanium trialuminide using ECAP routes A and Bc, both shape and distribution of Al₃Ti were changed by ECAP process, but they observed that wear property of the samples processed with different passes under different routes were quite similar. They assumed that this could be due to wear property of ECAP processed Al-Al₃Ti mostly depended on Al₃Ti the shape. Similar studies on Al/Al₃Zr alloy has likewise demonstrated that change of processing routs had little impact on wear behavior [174].

Kato et al. [178] conducted dry sliding wear tests on commercially pure titanium subjected to eight and twelve ECAP passes using route E. Route E is a combination two pressings using route C, followed by a 90° rotation before the third pressing and then another two pressing using route C [176]. This ECAP processing route led to an appreciable increase in strength with sufficient ductility, but did not bring about any noticeable improvement in wear behaviour.
Till now, limited works has been performed on wear performance of ultrafine grained materials processed by SPD methods. Appendix A lists the wear tests results currently available for different ultra fine grained materials. Most of the wear tests show that SPD techniques cause reduction in wear resistance. It is also evident that severe plastic deformation did not have a consistent effect on wear behaviour of materials. It can be seen that SPD resulted in increase in the wear resistance in some cases and decrease in wear resistance in others or no significant effect.

Generally, when the wear process is dominated by abrasive wear mechanism, the improved strength of SPD mostly leads to a higher wear resistance, as the high hardness stops hard particles penetrating into the material [156-161] when adhesive wear is the dominating mechanism; the wear resistance is more related to ductility and structure stabilization. In this case, SPD processing mostly leads to a worse wear resistance [170][171] when oxidation wear is dominating, the wear rate is decided by the oxidation rate and the property of the mechanical layer, mostly the SPD-processed material has similar wear resistance with the as received material [178]. In some case, as the SPD-processed material has higher chemical activity which leads to a higher oxidation rate, the SPD processed material has worse wear resistance [167].

Normally, wear is not only dominated by a single wear mechanism but several ones. Therefore, it is extremely hard to simply forecast the affect of SPD on wear resistance, but have to examine the whole wear process and each wear mechanism carefully. Nevertheless, it is clear to see that the SPD processing surely changes the wear behavior of material, either in a positive or negative way. It is important to seek the possibility of using SPD processing to achieve a better mechanical property of materials, and a better wear resistance at the same time.

Many of the above literature content showed evidence of severe wear mechanism such as adhesive and delamination, which indicates that the SPD materials (often soft materials such as Al, Mg and Ti) are still not good enough for wear applications. Therefore, more research works are necessary for seeking an improved good wear performance of SPD materials.