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ALUMINIUM METAL MATRIX COMPOSITE UNDER THE INFLUENCE OF HIGH STRENGTH CARBIDE PARTICLES ADDITION

by

Sumesh Narayan

A thesis submitted in fulfillment of the requirements for the degree of Doctor of Philosophy

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June, 2016
Declaration of Originality

Statement by Author
I, Sumesh Narayan, hereby declare that the work presented in this thesis, is to the best of my knowledge and belief original, except as acknowledged in the text, and that the material has not been submitted previously, either in whole or in part, for a degree at this or any other institution.

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Acknowledgments

Many people have been involved in this research work to make this thesis possible. I would like to take this opportunity to thank all these helpful colleagues.

First and foremost, I would like to thank my project supervisor, Dr Ananthanarayanan Rajeshkannan for his constant encouragement, guidance, enthusiasm and support throughout this project.

Secondly, I wish to express my gratitude to the Faculty of Science, Technology and environment’s, Faculty Research Committee for funding this research project. I am very grateful to all the academic and technical staff members of the Division of Mechanical Engineering. Special thanks to Mr. Shiu Dayal, Mr. Ashneel Deo and Mr. Sanjay Singh for their assistance in fabricating the machines and helping me with the experimentation phase of this research.

Finally, I would like to thank my parents for their continuous support and encouragement. I thank my wife for her patience, support and encouragement, especially those moments when the research project has taken most of my time.
Contributions and Publications


Abstract

The purpose of this thesis is to investigate the workability behavior of aluminium metal matrix composites prepared by powder metallurgy manufacturing route. The performance of carbide reinforced aluminium metal matrix composites (MMC’s) is studied in this investigation. The selected carbides in this study are titanium carbide, iron carbide, molybdenum carbide and tungsten carbide. Powder metallurgy manufacturing process is more ecological than many other industries, as it does not releases harmful gasses and pollutants in the atmosphere and uses re-cycled materials. However, one of the main concerns of this manufacturing route is the porosity left after the sintering process seriously affecting the strength of the material.

Complete experimental study on the densification, workability behavior and forming limit of powder metallurgy preforms of pure aluminium, Al-1TiC, Al-2TiC, Al-3TiC, Al-4TiC, Al-1Fe₃C, Al-2Fe₃C, Al-4Fe₃C, Al-6Fe₃C, Al-1Mo₂C, Al-2Mo₂C, Al-3Mo₂C, Al-4Mo₂C, Al-1WC, Al-2WC, Al-3WC and Al-4WC were carried out. Powder preforms having initial relative densities of 0.82 and 0.86, with three height-to-diameter ratios (aspect ratios) were prepared using a suitable die–set assembly on a 1 MN capacity hydraulic press. Sintering operation was carried in an electric muffle furnace at the temperature of 594 °C for a holding period of one hour. Three aspect ratios of 0.2, 0.4 and 0.6 were chosen for this research and the above mentioned powder metallurgy sintered aluminium preforms were machined to respective height-to-diameter ratio. Hot upsetting was carried out at the sintering temperature immediately after the sintering process and the forming process was stopped once visible cracks were seen on the free surface. Flat dies on the upper and lower surface were employed under dry friction conditions during hot upsetting.

The hot densification mechanism in forming of aluminium metal matrix composites is developed. The effect of carbide, its concentrations, preform geometry and initial relative density on the aluminium metal matrix composite’s densification during hot deformation is evaluated and presented in this work.
Workability characteristics of the aforementioned sintered powder metallurgy aluminium composites is established by studying under triaxial stress state condition the behaviour of densification, axial stress, hoop stress, hydrostatic stress, effective stress and formability stress index against axial strain. Further, attained density is considered to establish formability stress index and various stress ratio parameters behavior. Further, the influence of preform geometry and initial relative density on the workability behavior was analyzed and presented in this research work. It was found that the amount of carbide particles in the composite material shows significant effect on the relative density, respective stresses, workability and the formability stress index.

An efficient way to find the workability limit for powder metallurgy parts has been suggested. Oyane’s fracture principle was used to develop a theory to study powder metallurgy compacts. A least square technique was used to determine the constants in fracture criteria and these equations finally used to find workability limit. It is found that the projected technique was well in agreement with the experimental values. Further, the hot formability behavior of aluminium metal matrix composites using two key strain hardening parameters are studied to determining the failure zone.

Further, a galvanostatic pulse technique was used to determine the corrosion behavior of sinter-forged aluminium composites alongside microstructure studies to expose corrosion dynamics and presented in this report. It is strongly noted that this technique can be successfully used for such studies.

**Keywords**
Powder metallurgy; Aluminium metal matrix; Forming limit; Workability; Corrosion; Galvanostatic pulse technique; Densification; Workability limit diagram.
Nomenclature

$\varepsilon_\theta$  True hoop strain
$\varepsilon_z$  True axial strain or true height strain
$\varepsilon'_z$  True axial strain to fracture
$\varepsilon_{\text{eff}}$  Effective strain
$J_1$  First invariant of the stress tensor
$J_2'$  Second invariant of the stress deviator
$Y_0$  Yield strength of a solid material, Pa.
$Y$  Yield strength of a partially dense material, Pa.
$\alpha$  Poisson’s ratio
$de_\theta$  Plastic hoop strain increment
$de_z$  Plastic axial strain increment
$R$  Fractional theoretical density or relative density
$R_0$  Initial fractional theoretical density
$\sigma_z$  Axial stress, Pa
$\sigma_\theta$  Hoop stress, Pa
$\sigma_r$  Radial stress, Pa
$\sigma_m$  Hydrostatic stress, Pa
$\sigma_{\text{eff}}$  Effective stress, Pa
$\beta$  Stress formability factor
$\beta'_f$  Stress formability factor at fracture
$K$  Strength co-efficient
$K_i$  Instantaneous strength co-efficient
$n$  Strain hardening exponent
$n_i$  Instantaneous strain hardening exponent
$h_o$  Initial height
$h_f$  Forged height
$D_o$  Initial diameter

$D_f$  Forged contact diameter

$\beta_c$  $\beta_c$-parameter (pitting corrosion intensity)
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Chapter 1 Introduction

1.1 Overview

One of the serious issues faced by many small island developing states in the Pacific is the management of solid wastes. The amount of waste produced by factories and individuals are increasing day by day and soon will become a major problem (Kumar et al. 2007). Apart from other types of wastes, metal wastes such as light weight and low volume scrap iron & aluminium is a growing concern. Special consideration needs to be made sooner to prevent rapid filling of landfills by metals. There are several problems associated by this as well.

Scientific and technological tools are now slowly being used to contribute towards sustainable development. Powder metallurgy process is successfully used in several places to recycle these light weight and low volume scrap iron & aluminium. Same technology can be adapted for the small island developing states in the Pacific (Mihelcic, Zimmerman & Ramaswami 2007; Arena, Mastellone & Perugini 2003; Chung & Poon 1996).

The domestic solid waste production in the capital of Fiji, Suva, will soon become as big as in developed countries as one kg of domestic solid waste produced per day per capita. With economic development and modernization, today’s solid waste contains more and more industrial products and materials such as glass, metals, plastics and hazardous substances. Apart from the waste management for small island developing states in the Pacific, a greater problem exists by global warming and climate change which needs to be addressed (Arena, Mastellone & Perugini 2003; Chung & Poon 1996; Anastas & Zimmerman 2003).

Today, many researchers are working on producing frontier materials in this research field that will benefit our society in one way or another. Technological advancement and improved materials in terms of cost, manufacturability and strength are not the only concern today unlike in the traditional days. One of the most important issues today is
global warming, climate change issues and ecological and for any developments in technology and materials there is need to capture these important issues. One of the manufacturing routes so called powder metallurgy manufacturing route is considered to be green materials processing route (Mascarenhas 2004) in comparison to traditional or conventional casting materials processing route. Some of the merits of powder metallurgy manufacturing route over traditional casting process are that energy consumption is reduced by 50%, very little fumes & chemicals emitted in the atmosphere, use of recycled metals, increasing sales for green products, and growing disposal costs due to shortage of land fields (Mascarenhas 2004; Metal Powder Industries Federation 2012). Nippon steel reported in their technical report of several benefits of employing powder metallurgy technology in comparison to other manufacturing processes of producing titanium alloy parts. The production of titanium alloy parts with numerous near net shape technologies has been studied as answers to their high manufacturing cost, and stated strongly that powder metallurgy is one of the powerful cost reductions approaches (Nippon steel technical report 2002). The authors have showed further improvements within powder metallurgy manufacturing process by replacing the rubber mold used with thermo-plastic resin mold, optimizing the powder size distribution, addition of B$_4$C and Y$_2$O$_3$ and refining microstructures in an as-sintered condition resulting in excellent mechanical properties.

A report published by Metal Powder Industries Federation (2012) showed that a notch segment for truck transmission part consumed 2.847 kWh/piece when produced by original manufacturing process while it consumed 1.243 kWh/piece when produced by powder metal process as shown in Fig. 1.1.
Figure 1.1: Truck-Transmission Notch Segment Manufacturing Steps (Metal Powder Industries Federation 2012)

Every manufacturing process has its own merits and demerits and one of the main concerns with powder metallurgy processing route is that it produces parts with residual porosity. Hence, high strength applications of powder metallurgy parts are limited in the practical world. Secondary operations such as pressing or repressing, powder extrusion, powder rolling and infiltration can be employed to increase the strength of the powder metallurgy materials by eliminating the residual porosity (German 1994).

There are industries in the small island developing states in the South Pacific manufacturing parts using the traditional casting process. The casting process is costly, time consuming and produces parts which needs secondary material removal operations
to make the final product. Powder metallurgy is a solution to these problems as it produces parts to near net shapes. Although recycling can be done in a regular metal casting route but again the waste obtained by this route is unavoidable. A powder metallurgy process is proposed as it produces parts with no or very little wastes.

During the forming process the residual porosity left in the powder metallurgy parts after primary powder metallurgy process acts as stress concentration and appears as cracks on the free surface of the forming part. These cracks can be arrested by carefully designing the forming processes and die/mold designs. This means that the re-pressing of the free surface should be initiated before the cracks appear on the free surface. Therefore, an important study in forming of powder metallurgy parts is formability or many times referred to as workability studies. Further, Narayan and Rajeshkannan (2011; 2012) presented that the initial relative density, powder composition and preform geometry of the powder metallurgy parts are also important apart from other influencing parameters during the forming process as it influences the formability of the powder metallurgy parts. The present investigation by the authors is to analyze the workability of the aluminium metal matrix composites and how some of the important parameters stated above influence the workability and strength of the materials through hot deformation process. Usually the forming process is completed with just one blow of the hammer on the compact placed inside a die. This is more appropriate and efficient in industry when hot deformation is employed. Many studies (Narayan & Rajeshkannan 2011; James & West 2002; Esswein, Arrieche & Schaeffer 2008; Shanmugasundaram & Chandramouli 2009) found mainly employ cold upsetting over hot upsetting due to the hazard present during hot upsetting operations. The present investigations employs hot upsetting of aluminium plus hard carbide particles for workability, densification, deformation and corrosion analysis.

1.2 Powder metallurgy process

The powder metallurgy industries is growing fast in comparison to those using conventional process due to its added benefits and is considered to be eco-friendly manufacturing process when compared to conventional manufacturing route. Powder
metallurgical processing produces materials of extremely fine and uniform microstructure and enables the formulation of materials composed from different constituents yielding unique property combinations (Pokorska 2008). Further, a close control on the porosity level of some parts can be maintained using powder metallurgy manufacturing route which is not possible by other manufacturing processes. There is wide application of powder metallurgy parts in industry such as power plants, aircraft and naval industries, bearing materials, sintered friction materials for small wind turbines parts, cam shaft pulleys, gears, sprockets, connecting rods, nozzles, pump parts, offshore, energy and medical (Narayan & Rajeshkannan 2011; James & West 2002; Esswein, Arrieche & Schaeffer 2008; Shanmugasundaram & Chandramouli 2009; Pokorska 2008).

The starting material is the metallic powders in the conventional powder metallurgy manufacturing process. There are three steps in the conventional, powder metallurgy process as: (1) blending and mixing, (2) powder compaction in the required shape, and (3) sintering (Groover 1996) as shown in Fig. 1.2.

The elemental powders are mixed and blended homogenously before the compaction process to prepare an alloy or composite combination. The powder metallurgy technology is conducive nearly any material that can be processed in powder form. This technology is sometimes the only manufacturing method used to produce parts using materials such as porous materials, composite materials, refractory materials and special high duty alloys (Rosochowski, Beltrando & Navarro 1998). Quite often, powder metallurgy technique is used for material systems that are difficult to machine and difficult or impossible to cast such as parts require controlled porosity for use in filtration applications for air and water.

After the mixing and blending process, the required amount of powders are taken for the compaction process where high pressure is applied by the opposing punches squeezing powders contained in a die to form the powders into the required shape. A schematic of a single end die compaction method is shown in Fig. 1.3.
Figure 1.2: Powder metallurgy components: production cycle.

Figure 1.3: Single end die compaction method
After the compaction process the compacts are called the green compacts as it is low in strength, however, enough to be handled during the sintering process. Sintering is the process whereby powder compacts are heated so that neighboring particles fuse together, thus resulting in a solid object with better mechanical strength compared to the green powder compact.

A choice of atmospheres, including vacuum, are used to sinter different materials depending on their chemical compositions. As an example, precise atmosphere control allows iron/carbon materials to be produced with specific carbon compositions and mechanical properties.

Powder metallurgy metal matrix composites are extensively employed in industries due to some very important properties such as light weight, good mechanical properties such as fatigue, tensile, impact, rigidity (Kok 2005) and good chemical properties such as corrosion resistance (Torres et al. 2002). These components are commonly used in growing automotive, structural and aerospace industries (Davis 2012; Gururaja & Rao 2012). Further, powder metallurgy manufacturing route is used for mass production of precision engineering materials as its ability to produce complex parts with close tolerances and with maximum material utilization. Powder metallurgy is near net shape or net shape production technology and hence, there is no wastage or raw materials in comparison to other manufacturing route. It has proved to be cost effective of producing many parts such as porous materials, composite materials, refractory materials and special high duty alloys (Poshal & Ganesan 2009; Abouelmagd 2004; Rosochowski, Beltrando & Navarro 1998) to be used aircraft, automotive, and manufacturing industry. Further, powder metallurgy route is green manufacturing and energy efficient manufacturing then casting operation (Mascarenhas 2004).

Every manufacturing process has its advantage and disadvantage and so does powder metallurgy manufacturing route. Apart from many merits of this powder metallurgy route, one of the major drawbacks of this process is the residual porosity left in the compacts after the primary powder metallurgy process, blending and mixing, compaction
and sintering process. Hence, for high strength industrial applications the compacts needs to further go through the additional processes such as pressing, re-pressing, infiltration, powder rolling, extrusion, etc. to improve its mechanical properties (Raj et al. 2013; Rajeshkannan & Narayan 2009; Narayanasamy, Anandakrishnan & Pandey 2008). Fundamentally, two distinct methods of powder preform forging are carried out. Firstly, the powder is compacted without allowing any chance for inducing any change in the cross section termed as the repressive approach, significantly increasing the density with no lateral deformation. The other one involving compression of comparatively simple shaped compacts into intricate parts called as true forging. In this second approach significant increase in density is achieved with shape change occurring at the same time through massive plastic deformations together with significant flow of material in the lateral direction (Kuhn & Downey 1974; Tewari & Saran 1985). The plastic deformation of powder metallurgy material is similar to that of wrought (pore free) materials, but the analysis is complex due to the presence of substantial volume fraction of voids. The theories and techniques of analysis developed for studying problems in conventional metal forming methods is not applicable to powder metallurgy process as change of volume and yielding of void powder metallurgy materials are sensitive to the hydrostatic stress imposed. The way of deformation is relatively different in powder metallurgy materials in comparison to pore free materials and is a function of both density and the hydrostatic stress, which are enhanced during powder preform forging as strain increase (Jha & Kumar 1983).

Strength of particulate reinforced aluminium MMC’s can be enhanced by strain hardening phenomenon, grain boundary strengthening, solid solution strengthening, precipitation hardening and dispersion hardening. Strain hardening occurs with increasing interactions between dislocations reducing the dislocations mobility enhancing strength in materials. Grain refinements occurs by pinning of dislocations at the grain boundaries in turn enhancing strength and hardness in aluminium MMC’s. Further, in solid solution strengthening the dislocations are restricted by solute atoms and in precipitation hardening the precipitates restricts the dislocation movement in turn strengthening the aluminium MMC’s. Finally, in dispersion strengthening the particles restrict the
dislocation motion and this creates dislocation loops around the particles (Knowles et al. 2014; Hertzberg 1996).

1.3 Previous work and problem formulation

The powder metallurgy associated study has recently started at the Mechanical Engineering Department of The University of the South Pacific. Majority of the work carried out previously related to waste management possibilities for the pacific small island countries using various methods of which powder metallurgy were one. Kumar et al. (2007) proposed possible use of powder metallurgy process for the Fiji Islands to solve the ongoing problem of significant waste production. Further, study was also done on the production of metal powders from waste materials. There has been no work conducted on the primary and secondary processing of the powder metallurgy parts at the department which motivated me to carry out this research.

Nowadays, numerous investigators are working on producing frontier materials that will benefit our society in one way or another. Aluminium MMC’s are in demand for industrial applications due to some very good properties main one being high strength to density ratio (Derakhshandeh & Jahromi 2011; Sahin 2003). Carbide reinforced aluminium are widely used as carbide particulate are good in wear and corrosion resistance, high strength and hardness. TiC, SiC and Al₂O₃ reinforced aluminium parts are extensively employed in industry due to many good properties such as low specific density, good wear resistance and low thermal expansion coefficient. SiC reinforced aluminium composites (Sahin 2003; Eslamian, Rak & Ashgriz 2008), TiC reinforced aluminium composites (Narayanasamy, Senthilkumar & Pandey 2007) and Al₂O₃ reinforced aluminium composites (Zhang et al. 2009) have shown improved properties in aluminium metal matrix composites such as hardness, tool wear in turning, improvement in wear resistance and reduced corrosion rate. In this research the authors studied reinforcing aluminium metal matrix composites with WC, TiC, Fe₃C and Mo₂C due to its possible industrial uses. Even though many literature deals with silicon carbides based aluminium composites, the full analysis such as mechanical properties, microstructure, workability, fractography are not attempted via hot deformation. Further, little work has
been attempted via hot deformation on titanium carbide, tungsten carbide, iron carbide and molybdenum carbide to understand its behavior (Eslamian, Rak & Ashgriz 2008; Aigbodion & Hassan 2007; Wang et al. 2008; Izadi et al. 2013; Sheibani & Fazel 2007; Zhong et al. 2011; Lui et al. 2013). WC, Fe3C and Mo2C are rarely found in literature as reinforcing particulates; however, the authors believe that these particulates may provide enhanced properties when reinforced in aluminium metal matrix composites and is great significance for industrial applications. Hence, in this research work several aluminum MMC’s are prepared using powder metallurgy process to investigate the hot workability behavior.

1.4 Project objectives
To produce the powder metallurgy products of required shape and size or even to do any fundamental research on the development of any frontier materials for the betterment of society, apart from many other parameters been as influencing phenomena, one of the fundamental quest is to produce powder metallurgy parts with free of defects. The workability or formability of the powder metallurgy material plays a major role in determining if the powder metallurgy material will be formed successfully or fracture initiates in the forming process. Workability gives the measure of the deformation limit prior to material failure during the deformation process and is particularly important to study while powder metallurgy preforms are subject to secondary deformation. During secondary deformation powder metallurgy preforms has considerable material flow in the lateral direction due to induced height strain. During lateral flow of material or deformation, apart from pore closing mechanism, the pores also elongate in the lateral direction. Workability characteristics depend on the material, forming stress and strain rate, porosity, forming friction and forming temperature (Narayanasamy, Ramesh & Pandey 2005; Rahman & El-Sheikh 1995; Narayanasamy, Anandakrishnan & Pandey 2008). Hence the following objectives are foundations for the research.

1. Establish deformation-densification behavior;
2. Establish workability behavior of the selected composition; and
3. Establish corrosion behavior of the selected composition.
Chapter 2  Literature Review

2.1  Overview
Powder forging have been studied widely and raising much interest in many parts of the industry as an economic method of producing high strength, high ductility parts from metal powders (Poshal & Ganesan 2009) as powder metallurgy method competes with other methods on the basis of cost which can be lower for high volume production of complicated components. Powder metallurgy components are widely used in sophisticated industrial applications while the worldwide popularity lies in the ability of powder metallurgy technique to produce complex shapes with exact or very close tolerance at a very high production rate at minimal costs (Kandavel, Chandramouli & Ravichandran 2010; Wang & Zhang 2008). The huge application of ferrous powder metallurgy material in automotive and aerospace industry provides reasons for researchers to analyze powder metallurgy materials behaviour under metal forming processes (Lindskog 2003). Powder preform forging involves the fabrication of a preform by primary deformation processing technique, followed by the secondary pressing of the preform to its final shape with substantial densification. Sintered powder metallurgy compacts are made by the process of compacting and sintering ferrous powder and non-metal powder.

A known limitation of this route is the large number of small voids left in components after sintering. Plastic deformation is a main way to improve the performance of sintered ferrous material and obtain the final product. In general the preform produced by the conventional process will undergo so large degree of plastic deformation with enhanced level of densification (Hua et al. 2006; Liu et al. 2006). Though plastic deformations of powder preforms are similar to that of conventional fully dense material, the additional complications are because of substantial amount of void fractions. Because there is a large number of residual porosity in the sintered powder materials, plastic volume change of sintered compacts will result from the void reducing and closing during plastic deformation. Hence, the endeavor of the present researchers being producing parts to near theoretical density; however 100 percent dense component cannot be produced.
2.2 Aluminium metal matrix composite

Aluminium metal matrix composites are rapidly growing as it is able to satisfy the recent demands of advanced engineering materials. Aluminum metal matrix composites are used for wide variety of industrial applications due to its unique properties such as improved mechanical properties, possible reduction in manufacturing costs, improved corrosion, low specific density, high strength, low thermal expansion, good wear resistance, possible and positive enhancement in properties with increased industrial applications and are economic viable (Sahin 2003, 1998; Derakhshandeh & Jahromi 2011; Zebarjad & Sajadi 2007; Fogagnolo et al. 2003). The performance of aluminium metal matrix composites greatly depends on the reinforcing particulate and further the manufacturing technique also depends on the reinforcing particulates to some extent. Several combinations of single reinforcing and hybrid reinforcing particulate are tested and presented in research for industrial use. This work tries to review the different combinations of reinforcing particulates in manufacturing of aluminium metal matrix composites, the performance of these materials and fabrication techniques involve in the manufacture of these materials.

Any development in the properties of existing aluminium materials will boost the aluminium industry significantly. Improved mechanical, chemical and physical properties that cannot be attained by classical ingot method can be achieved by powder metallurgy manufacturing route (Khairaldien, Khalil & Bayoumi 2007; Das et al. 2010). Ductile aluminum matrix reinforced with stronger and stiffer carbides provides a combination of properties of the metallic material and ceramic reinforcement components (Prabhu et al. 2006). A good choice of alloy composition makes it possible to produce parts with suitable density, thermal expansion, wear and corrosion resistance, good combinations of strength, ductility and toughness, processing flexibility and homogenous microstructure (Derakhshandeh & Jahromi 2011; Sahin 2003; Narayanasamy, Senthilkumar & Pandey 2007). Further, reinforcements help in high temperature applications of low density metals such as aluminium. Aluminium metal matrix composites (MMC’s) have taken over conventional metallic alloys as their use have extended in more industrial applications such as marine, automobile, aerospace, defense, sports and recreation.
industries (Das et al. 2014). Aluminium has become the most used metallic alloy in the industry of MMC’s production due to valuable reasons as highlighted in (Casati & Vedani 2014; Prasad & Krishna 2011; Liu et al. 2009).

Metal matrix composites can be categorized depending on the reinforcement type. Three different categories are as follows: (1) Fiber reinforced MMC’s, (2) Particulate reinforced MMC’s and (3) Whisker reinforced MMC’s. Fiber reinforced MMC’s have reinforcements in fiber form while particulate reinforced MMC’s have reinforcements in powder form. Whisker reinforced MMC’s have proved to have highest mechanical properties, however, have health hazards limiting its industrial use. Based on industrial application and manufacturing ease, particulate reinforced MMC’s proves to hold upper hand over fiber reinforced MMC’s and whisker reinforced MMC’s. The advantages of using particulate reinforced aluminium metal matrix composites over whisker or fiber reinforced aluminium metal matrix composites are reduced health hazards, manufacturing ease, lower manufacturing costs, better forming ability, superior properties at high temperature, improved wear resistance and many more as highlighted in the following work (Surappa 2003; Sirahbizu, Mahapatra & Jha 2013; Kok 2005). Generally the properties are more isotropic when compare to fiber and whisker reinforced MMC’s.

It is also noted that limited supply of the reinforcement particulate especially in the developing countries is a major drawback with poor ductility, low fracture toughness and unpredictable corrosion behavior (Kok 2005; Bhandakkar, Prasad & Sastry 2014). Many researches are directed towards the selection of the appropriate reinforcing particulate to solve such problems however, there is no clear cut procedure to do so. This clearly points to the fact that reinforcing material has a significant output on the performance of the final aluminium metal matrix composite produced. However, apart from this there are any other factors that influence the performance of aluminium metal matrix composites (Liu et al. 2006; Narayanasamy, Ramesh & Pandey 2006). The researchers and authors have generally used the three approaches in the selection of the appropriate reinforcing particulate in the manufacturing of aluminium metal matrix composites (Narayanasamy,
Firstly, finding the low cost and alternative reinforcing particulate that can replace high cost and limited availability of conventional reinforcing materials. The application of agro-industrial waste in aluminum metal matrix composites has been investigated by many authors (Loh et al. 2013; Anilkumar, Hebbar & Ravishankar 2011; Aigbodion et al. 2010; Alaneme et al. 2013; Lancaster, Lung & Sujan 2013; Rohatgi, Gupta & Alaraj 2006) due to low cost of reinforcement materials and chance of reducing environment pollutants. It was reported (Loh et al. 2013; Anilkumar, Hebbar & Ravishankar 2011; Aigbodion et al. 2010) that the used of agro-industrial waste showed promising improvements in the properties of the composite developed over unreinforced alloy. However, they produced much inferior properties when compared to conventional reinforcing particulate aluminium composites (Prasad & Krishna 2011; Alaneme et al. 2013). Reference (Lancaster, Lung & Sujan 2013) reported that the corrosion behavior of the base metal, aluminium, was far much better than the aluminium fly ash metal matrix composite. The percentage of fly ash was varied from 5 wt. % to 15 wt. % and the materials were prepared through powder metallurgy technology. The authors presented that the addition of fly ash particles in the aluminium matrix composite gave rise to pit initiation sites accelerating the corrosion behavior. Reference (Rohatgi, Gupta & Alaraj 2006) reported on the tensile strength of fly ash reinforced aluminium composites. The authors clearly projected that the addition of fly ash to the aluminum matrix composite decreased the tensile strength significantly over unreinforced aluminium alloy. On the other hand reference (Ramachandra & Radhakrishna 2005) highlighted that the aluminium matrix composite containing fly ash as reinforcing particulate produced good density over unreinforced aluminium alloy. However, they also highlighted that there was a significant decreasing in the final density of the part with increasing fly ash reinforcement particulates. Reference (Apasi et al. 2012) studied the wear behavior of coconut shell ash/composite and reported that the wear rate decreased with an increase in the weight percentage of coconut shell ash particles. Generally the use of agro-industrial waste alone in the development of advanced aluminium matrix composite are limited and
more work is required to successfully utilize these products. Further, appropriate methods to manufacture these materials are still investigated. Recently, agro-industrial wastes are used in the manufacture of hybrid aluminium matrix composites usually as secondary reinforcing particulate. Aluminium metal matrix composites are widely used in the automobile and aircraft industry for engine parts as it can reduce the overall weight, fuel consumption and pollution (Singh & Chauhan 2015). The reinforcement particles used for these applications are usually silicon carbide or alumina. Silicon carbide or alumina are denser than the aluminium alloy and in turn increases the weight of the aluminium composite depending on the reinforcement content with other concerns of machining due to increased hardness. Such problems can be solved by introducing the secondary reinforcement particulates in the form of agro-industrial wastes; however, more work is required in this research field to obtain the composites with good mechanical properties (Safiuddin et al. 2010; Alaneme, Tolulope & Peter 2014; Alidokht et al. 2011).

The second approach in the attempt to improve mechanical properties of aluminium metal matrix composites is by reducing the particle size of the reinforcing particles from micro-scale to nano-scale. In this approach the particles size are usually maintained between $< 50 \mu m$ to an average of $< 100 \text{ nm}$ (Knowles et al. 2014; Prabhu et al. 2006). Reference (Mobasherpour, Tofigh & Ebrahim 2013; Poovazhagan et al. 2013; Boostani et al. 2015) show that there are improvements in the mechanical properties of aluminium metal matrix composites when reducing the size of the reinforcing particulate from micro-scale to nano-scale. However, some of the negative points of using nano-sized reinforcing particulates such as high cost and availability of nano-particles, high propensity of nano-particles to agglomerate, low wettability and recently developed put limitations on the use of nano-sized reinforcing particulates in the production of aluminium metal matrix composites (Casati & Vedani 2014; Boostani et al. 2015; Rana, Purohit & Das 2012).

The third approach involves the development of aluminium matrix composite using more than one reinforcing particulate, so called hybrid composites. Some authors have reported improvements in the mechanical properties of using hybrid composites over single
reinforced aluminium matrix composites (Gheorghe et al. 2015; Sharifi & Karimzadeh 2011; Alaneme & Olubambi 2013). Other reported that some of the single reinforced particulate aluminium metal matrix composites produced better properties for advanced engineering applications (Narayanasamy, Ramesh & Prabhakar 2009; Krasnowski, Gierlotka & Kulik 2015; Izadi et al. 2013). The development of hybrid aluminium metal matrix composites is on the rise in search of developing high performance – low cost materials. Several manufacturing techniques are tested to optimize between high performance and cost. Recent developments in the use of agro-industrial waste as secondary reinforcements have been popular in recent years.

2.3 Reinforcing materials in aluminium metal matrix composites

It is known that the final property of the aluminum matrix composite depends on the reinforcing particulates and which further governs the processing techniques used to produce these composites. Hence it is noted that the reinforcing particulate type, size, shape, modulus of elasticity, hardness, morphology, percentage of volume fraction, distribution in the matrix are important and efficiency of bonding between the matrix/reinforcing phase (Das et al. 2014; Alaneme & Aluko 2012). Depending on the specific application the reinforcements in aluminium MMC’s can be continuous (fibers) or discontinuous (short fibers, particulates and whiskers). The reinforcements in aluminium MMC’s found are ceramics, mostly oxides, carbides and nitrides. Generally these are silicon carbide and alumina, however, titanium carbide, tungsten carbide, boron carbide, boron and graphite are also common (McKimpson & Scott 1989; Terry & Jones 1990). It is also noted that finer reinforcing particulate produces strong interfacial bonds and higher strength composites compared to bigger particle size.

Silicon carbide (SiC) particulates are the most common discontinuous reinforcement used in reinforcing aluminium alloy due to their compatibility. SiC reinforced aluminium MMC’s are used where modulus requirements are as high as steel, however, with reduced strength requirements. Reference (Taha, El-Mahallawy & El-Sabbagh 2008) studied workability of aluminium SiC and Al₂O₃ reinforced metal matrix composites. They concluded that workability is affected positively by the following, decreasing particulate
weight volume percentage, decreasing particulate size and reinforcing with SiC in a wrought alloy matrix rather than Al$_2$O$_3$. Reference (Narayanasamy, Ramesh & Prabhakar 2009) carried out experimental investigations on workability behavior of Al-SiC showing the effect of particle size of SiC. SiC content was varied from 0% to 20% with different particle size of 50, 65 and 120 $\mu$m. They showed that as the SiC content increases the pore size becomes smaller in tend increases the formability stress index. For preforms with higher percentage addition of SiC the crack initiation starts with lower fracture strain. The stress ratio parameter and formability stress index is found to be higher due to better densification of Al-SiC composite when compared to pure aluminium. Martin, Forn and Nogue (2003) studied the strain hardening behavior and temperature effect on Al-2124/SiC in an attempt to optimize the parameters for deforming processes of these materials. It was clearly noted that the addition of SiC particulates to aluminium alloy increases the strengthening properties such as elastic modulus but decreases the failure strain and strain hardening exponent due to true plastic localization starting. Reference (Evans & Shariff 2004) reported that the porosity in cast 20 wt.% aluminum MMC had a dominant influence on the fatigue performance. On the other hand, the 25 wt.% SiC aluminium composite by powder metallurgy technique, with finer reinforcement size, performed in a similar way to the conventional alloy.

Al$_2$O$_3$ is another common reinforcement of aluminium alloys because of its oxidation resistance and chemical inertness comparative to aluminium. Al$_2$O$_3$/Al is used for its high strength and modulus. Reference (Narayanasamy, Anandakrishnan & Pandey 2008) analyzed the densification and workability behavior experimentally by studying the effect of geometric and matrix worked hardening. Another set of specimen was subjected to the annealing process at 200 °C for 30 mins after every step of deformation to study on the effect of geometric work hardening. Reference (Zuhair & Amro 2002) presented the corrosion behavior of powder metallurgy aluminium alloy Al$_2$O$_3$ MMC. Some of the drawbacks found were the number of pit initiation sites increased with increasing volume fraction of the reinforcements and sub-micron alumina reinforcement agglomerate and form clusters which were observed in the microstructures. Reference (Al-Dheylan & Hafeez 2006) studied the damage and failure processes caused by tensile loading on
Al₂O₃ reinforced aluminium MMC. Test results showed significant improvements in the modulus of elasticity and tensile strength with increasing volume fraction of the reinforcements. Dobrzański, Wlodarczyk & Adamiak (2005) studied the structure, properties and corrosion behavior of aluminium alloy reinforced with Al₂O₃ particles. It is seen that hardness increased by 29% for 15 wt.% Al₂O₃ reinforced aluminium composite when compared to unreinforced aluminium alloy, however, it is seen that ultimate compressive strength decreased with increasing Al₂O₃ reinforcements. Further, results indicated that the composite materials reinforced with 5 and 10 wt.% Al₂O₃ produced higher corrosion resistance compared to the matrix material, whereas for 15 wt.% Al₂O₃ had the worst resistance.

Recently titanium carbide (TiC) reinforced aluminium metal matrix composites are studied for its potential use. Titanium carbide based MMC’s are widely used where wear and corrosion are the main mode of failure. Titanium carbide particulates are extremely hard and the improvement in wear resistance and reduced corrosion rate is expected (Narayanasamy, Senthilkumar & Pandey 2007). TiC based composites with nickel alloys and iron alloys are presently used in high strength uses (Narayanasamy, Senthilkumar & Pandey 2007; Butuc, Gracio & Rocha 2006). Mohapatra et al. (2015) exposed the microstructure and mechanical properties of Al–TiC composites by hot consolidation technique. Improved Young’s modulus and mechanical properties with significant ductility were seen in the composites which demonstrated the helpful use of TiC reinforcement. The authors concluded that Al–TiC aluminium composites prepared by this method are suitable for structural and industrial uses, like other aluminium based MMC’s. Sherif et al. (2015) produced Al-TiC composites in a high frequency induction heat furnace to study the corrosion properties in sodium chloride solutions. After the mixing process in an high energy ball milling machine the powders were subjected to compaction and sintering simultaneously at a pressure of 10 MPa and temperatures of 900 °C, 1100 °C and 1300 °C. They reported that Al–TiC composites produced the best resistance to corrosion at 900 °C followed by 1100 °C and the lowest resistance was found at 1300 °C. Ali et al. (2013) compared the wear and hardness properties of Al–TiC and Al–(TiC+Fe₃C+Fe₂Ti+Fe) composites produced by a new synthesis technique. The
wear and harness properties of the two composites produced were almost identical and it can be concluded that pure TiC in aluminum matrix composite is as good as synthesized powders containing TiC, Fe$_3$C, Fe$_2$Ti, and Fe as the reinforcement material. Rai et al. (2013) studied the forming behavior of TiC reinforced aluminium composites by comparing microstructure and mechanical properties of as-cast, forged and rolled specimens. A uniform distribution of the TiC particulates was observed in the forged and rolled specimen’s, reason for good tensile strength in these specimens. The primary samples were prepared by the reaction of aluminium with K$_2$TiF$_6$ and graphite powder at 1200 °C in an induction furnace. The specimens were than subjected to hot/cold rolling and forging. Same specimens were further tested for machinability and compared to Al–TiAl$_3$ composite and Al–Si alloys (Rai et al. 2006). The authors reported enhancement in the quality of the machined surface with increased amount of TiC particles in the composite together with lower cutting force compared to Al–TiAl$_3$ composite and Al–Si alloy. Tyagi (2005) reported significant reduction in wear rate and coefficient of friction of TiC reinforced aluminium composites compared to base material. On the other hand the corrosion behavior of TiC reinforced aluminium composites prepared by stir casting route was investigate by Murthy and Singh (2015). The microstructures and polarization investigations revealed an increase in corrosion resistance in composites compared to the matrix alloy due to excellent bond integrity of TiC particulates with aluminium and possible electrochemical decoupling between TiC particles and matrix alloy. Reference (Chaira, Sangal & Mishra 2007) fabricated Al-Fe$_3$C composite by mixing Fe$_3$C with aluminium powders, and then by hot pressing or cold compaction and sintering. Fe$_3$C particulate was first prepared by mechanical alloying in a specially built dual-drive planetary mill using iron and graphite powders. The microstructures of hot-pressed Fe$_3$C reinforced aluminium shows exceptional compatibility between aluminium matrix and Fe$_3$C particulates and can be successfully used to produce advanced engineering materials.

The interest in developing tungsten carbide (WC) based aluminum composite has been increasing significantly in recent years (Pydi, Adhithan & Bakrudeen 2013). Simon et al. (2015) fabricated 5-15 wt.% WC reinforced aluminium composites by powder metallurgy
route. Firstly a homogenization process was carried out to prepare the respective blends of powders in a planetary ball mill for 30 mins, which were then subjected to cold pressing at a pressure of 500 MPa. The green compacts were sintered at 580 °C for 20 mins. Increases in the micro-hardness and wear resistance were reported in the WC reinforced composite materials over aluminium alloy. A very similar fabrication technique was employed by Pydi, Adhithan & Bakrudeen (2013). The microstructural analysis showed uniform distribution of the WC particulates in the composites with good interfacial bonding between the matrix and tungsten carbide particles. The inclusion of tungsten carbide in the composite enhanced the impact resistance of the composite by reducing the cracks and voids in the crystal lattice. On the other hand, Amarnath and Sharma (2013) prepared 5-20 wt.% WC reinforced aluminium composites by liquid metallurgy route. The WC particles, preheated at 773 °C, were introduced in the molten base material with the help of vortex created by a stirrer rotating at 500 rpm. Selte and Ozkal (2015) studied the production and characterization of Al-WC composite powders via mechanical alloying. They reported successful production of Al-WC composite powders.

The authors believe that more work is required in the study of titanium carbide, iron carbide and tungsten carbide reinforced aluminium metal matrix composite. Analysis such as mechanical properties, microstructure, workability and fractography are rarely found in literature. The lack of research and development of TiC, Fe3C and WC reinforced aluminium metal matrix composites can be a good motivation to carry out such work in this area.

2.4 Fabrication of aluminium metal matrix composites

Narayanasamy, Ramesh and Prabhakar (2009) fabricated 20 wt.% SiC reinforced aluminium MMC by powder metallurgy technique with an average sintering time of 120 min at 600 °C. The compacting pressure applied was 518 MPa, which was maintained for all composition of SiC composites. SiC-Al powders were mixed in a pot mill for 2 hours to obtain homogenous mixture. The microstructures presented show a homogenous distribution of the SiC particles. Reference (Narayanasamy, Anandakrishnan & Pandey
2008) successfully fabricated 3.5 wt.% alumina reinforced aluminium MMC’s via powder metallurgy manufacturing route. The preforms were ceramic coated and dried before the sintering process to avoid oxidation. The preforms were sintered at 500 °C for 100 mins and ultimately cooled to room temperature in the furnace itself. Martin, Forn & Nogue (2003) fabricated 2124 aluminum alloy and 2124 aluminum alloy reinforced with 17 wt.% SiC particles with an average size of 1.4 \( \mu m \) via powder metallurgy process. The uniformity of distribution of SiC particles in the matrix material was confirmed by the optical microstructure and fine matrix grain size, with an average diameter of about 1 \( \mu m \) was seen in both materials. Kumar, Loganathan and Narayanasamy (2011) successfully fabricated 2 – 8 wt.% glass reinforced aluminium composite to study the formability characteristics using the conventional powder metallurgy route. The parts were then subjected to open die forging together with pure aluminium alloy prepared via the same technique. Microstructural examination was also carried out and presented showing the glass particles distribution and pore sizes. Reference (Dobrzanski, Wlodarczyk & Adamiak 2005) fabricated Al\(_2\)O\(_3\) reinforced aluminum composite by powder metallurgy route that involved blending of aluminium powder with 5, 10 and 15 wt. % Al\(_2\)O\(_3\) in vibratory ball mill to acquire homogenous distribution of reinforcement particles in the matrix material. The cold state compaction was carried out at 350 kN and then the compacts were sintered at 500 °C and finally extruded at this temperature at extrusion pressure of 500 kN. The microstructures revealed uniform distribution of the reinforcement particles in the matrix material.

Hung et al. (1995) manufactured 22 wt.% SiC reinforced aluminium composite by permanent mold casting procedure. The parameters chosen are stirring speed of 250 rpm, pouring temperature of 705 °C. At the same time the authors also fabricated 20 wt.% sintered powder metallurgy SiC/Al composites to study the machinability costs. They reported that irrespective of the cutting tool materials used, fractured SiC particulates and de-bonded matrix-reinforcement interface were seen on the surface. Similar fabrication technique, casting and powder metallurgy was used by reference (Evans & Shariff 2004) to study fatigue behavior. Reference (Zuhair & Amro 2002) fabricated varying 5 – 20 wt. % TiC reinforced aluminium composite by hot consolidation technique, the hot pressing
was carried out at a temperature of 400 °C with an applied pressure of 400 MPa for 5 min under a vacuum of 10⁻⁴ mbar. The field emission scanning electron micrographs exposes the homogeneous distribution of TiC particles in the aluminium matrix. Hamed, Shady and El-Desouky (2001) fabricated 10 wt.% SiC reinforced aluminum composite via ingot metallurgy using ultrasonic method to refine the grains. The comparatively low melting temperature together with small stirring time and high solidification rate resulted in composites having high strength with adequate ductility. Cocen and Onel (2002) prepared Al-5%Si-0.2%Mg alloy and SiC reinforced aluminium composite using melt stirring technique in an argon purged induction furnace. Naher, Brabazon and Looney (2004) prepared SiC reinforced aluminium composite using their own designed and constructed quick quench compocasting equipment’s equipped with temperature control sensors and reported that SiC can be successfully distributed in the matrix material by stirring in the semi-solid state without any wetting agent. The microstructures were presented showing the SiC particles distribution. Stir casting method to prepare aluminium MMC’s are also presented elsewhere (Kayal et al. 2011; Shehata et al. 2013). On the other hand Rajiv, Srinivasan and Balakrishnan (1997) prepared 10-30 wt. % SiC reinforced aluminium composites via powder metallurgy route by dry blending the aluminium and SiC particles which was pelletized into circular disc under a compacting pressure of 97 MPa. Further, the compacts were sintered at 580 °C in an inert atmosphere to avoid oxidation. Boopathi, Arulshri and Iyandurai (2013) produced SiC reinforced aluminium, fly ash reinforced aluminium and fly ash-SiC reinforced aluminium composites by stir casting technique. Umanath, Kumar and Selvamani (2013) fabricated SiC and Al₂O₃ reinforced aluminium composites using stir casting technique. Further, Kumar and Dhimen (2013) fabricated 7 wt.% SiC and 3 wt.% graphite reinforced aluminium composites and Babu and Krishnan (2012) prepared 10 wt.% SiC and 5 wt.% B₄C hybrid aluminium composites using stir casting technique. The following processing parameters were used (Boopathi, Arulshri & Iyandurai 2013; Umanath, Kumar & Selvamani 2013; Kumar and Dhimen 2013; Babu and Krishnan 2012). Firstly, pure aluminium was melted in an induction electric resistance furnace in a temperature in a range of 710-750 °C and stirred with a mechanical stirrer at a speed range of 300-600 rpm for 10-20 mins. The stirring created turbulence and vortex motion. Simultaneously the reinforcement materials are preheated
for 1-3 hours at the required temperatures to remove moisture which is introduced in the molten matrix alloy with the help of the vortex created by stirring. After complete addition of the reinforcement particles into the molten metal, the liquid composite is poured into steel mold preheated to respective temperatures in a range of 250-700 °C and allowed to solidify and cool in the atmosphere.

Dikici (2012) employed conventional hot pressing techniques in argon atmosphere to fabricated Al–Cu-based three-layered metal matrix composites reinforced with TiC particles. Falcon et al. (2011) fabricated 44 wt.% TiC reinforced Mg-Al composite by pressureless melt infiltration. Firstly, the Mg-Al powders were sintered in a controlled atmosphere of argon gas at 1250 °C for 60 mins and then infiltration was carried out for 12 mins at 950°C. A very similar fabrication technique, pressureless melt infiltration, is also employed by Albiter et al. (2006). Rajeshkannan and Sharma (2014) prepared hybrid composites of Al-2WC-4Fe3C, Al-2WC-8Fe3C and Al-2WC-12Fe3C by powder metallurgy route to study densification and corrosion behavior. Firstly, a dry blending process was carried out of aluminium powder with respective percentages of tungsten and iron carbide powders at 200 rpm for 2 hours, followed by compaction at around 350 MPa. Tong and Ghosh (2001) fabricated TiC reinforced aluminium composites using a novel technique firstly by ingot metallurgy and then by rapid solidification. Powders of Al, Ti and graphite were first melted in an induction furnace to the processing temperature of 1125 °C for 1 hour. Then the temperature was increased to 1325 °C and held for 10 mins before direct chill cast into ingot billets of 12mm in diameter. A blanket of argon gas was always present on top of the melt.

After broad literature review it is seen that for particulate reinforced aluminium metal matrix composites, powder metallurgy and molten metal stir casting techniques are considered most promising. Powder metallurgy route is classified under solid phase fabrication methods while stir casting technique is classified under liquid phase fabrication methods. Some important factors that need to be addressed in any processing techniques to produce parts with superior mechanical properties are as follows. (1) The processing route shall be able to produce wide varieties of matrix and reinforcements
materials with good reproducibility at minimum cost and maximum productivity, (2) The processing route shall be able to handle wide variety of reinforcement shape, size and volume fraction, (3) The uniform distribution of reinforcement particulates in the matrix alloy is highly desirable without any breakage and degradation, (4) good bonding strength between matrix alloy and reinforcement materials and (5) the final product shall have minimum porosity with good density. The two techniques have been summarized below.

2.4.1 Powder metallurgy
Powder metallurgy manufacturing process is becoming very important in the fabrication of aluminium metal matrix composites due to the following reasons, more volume fraction of reinforcements are possible, reinforcements with nano-sized particulates are possible and production of near-net shape products. Problems such as particle clustering, wettability and formation of unwanted phases are eliminated in the powder metallurgy processing route compared to liquid processing route (Khairaldien, Khalil & Bayoumi 2007; Das et al. 2010; Derakhshandeh & Jahromi 2011). A schematic diagram of the process is summarized in Fig. 1.2 and the processing steps are discussed below.

Step 1: Selection of matrix and reinforcement materials
Aluminium alloy of size 100-150 \( \mu m \) may be chosen to prepare the composite. Generally it is found that aluminium powder of size 150 \( \mu m \) is used by several authors (Sahin 2003; Narayanasamy, Senthilkumar & Pandey 2007; Mamatha, Pruthviraj & Ashok 2011). The sizes of common reinforcements vary within the wide range of 1-4200 \( \mu m \). Reference (Narayanasamy, Ramesh & Prabhakar 2009; Martin, Forn & Nogue 2003) used fine size of 1, 50, 65 and 120 \( \mu m \) SiC particles, whereas Jayaram and Biswas (1999) studied the effect of coarse particle size of 4200 \( \mu m \). Titanium carbide powders of 10 \( \mu m \) (Mohapatra et al. 2015), 48 \( \mu m \) (Narayanasamy, Senthilkumar & Pandey 2007) and 50 \( \mu m \) (Murthy & Singh 2015) were used to produce TiC reinforced aluminium MMC. Tungsten carbide powders of 1 \( \mu m \) (Simon et al. 2015), 50 \( \mu m \) (Rajeshkannan & Sharma 2014) and 8 \( \mu m \) (Selte & Ozkal 2015) were used to produce WC reinforced
aluminium MMC and a particle size of 50 \( \mu \text{m} \) was used to produce Fe\(_3\)C reinforced aluminium composites (Rajeshkannan & Sharma 2014). It is found that the reinforcement materials particle size of around 30-60 \( \mu \text{m} \) are generally employed in the fabrication of aluminium matrix composites.

**Step 2: Selection of volume fraction of reinforcement materials**

This can be selected depending on specific applications as the presence of the reinforcement produces changes in the microstructure of the matrix and therefore to the overall properties. The volume fraction of SiC generally found range from 2-30 wt.%. Reference (Mamatha, Pruthviraj & Ashok 2011) used 2, 4, 6 wt.% SiC to manufacture SiC reinforced aluminium composites, whereas reference (Sahin 2003; Narayanasamy, Ramesh & Prabhakar 2009; Rajiv, Srinivasan & Balakrishnan 1997) used higher SiC volume fraction of 10, 20 and 30 wt.% to fabricate SiC reinforced aluminium composites. Poovazhagan et al. (2013) used low weight fraction in a range of 0.5-1.5 % SiC to prepare aluminium composites. Tyagi (2005) used low weight fractions of TiC (0.07, 0.12 and 0.18 %), whereas Narayanasamy, Senthilkumar & Pandey (2007) used high weight fractions of 4 % to produce TiC reinforced aluminium composites. Reference (Zuhair & Amro 2002) fabricated varying 5–20 wt. % TiC reinforced aluminium composite by hot consolidation technique. The Tungsten Carbide particulate was added in proportions of 1, 2 and 3 wt.% to produce aluminium reinforced with tungsten carbide particulate and flyash metal matrix composites (Srikanth & Amarnath 2015).

**Step 3: Blending and mixing of matrix alloy and the reinforcement particulates**

The elemental powders are mixed and blended homogenously before the compaction process to prepare an alloy or composite combination. The powder metallurgy technology is conducive nearly any material that can be processed in powder form. Narayanasamy, Ramesh and Prabhakar (2009) homogenously mixed Al and SiC powders a planetary ball mill at 200 rpm for 2 hours. Similar steps were taken by reference (Narayanasamy, Anandakrishnan & Pandey 2008; Dobrzanski, Wlodarczyk & Adamiak 2005) to homogenously mix Al and Al\(_2\)O\(_3\) powders. Rajeshkannan and Sharma (2014) employed planetary ball mill machine to mix Al, WC and Fe\(_3\)C powders to prepare hybrid
composites. A uniform distribution of reinforcement particles in the matrix alloy was achieved in 2.5 hours at 200 rpm. The same can also be achieved by mechanical alloying process. High-energy ball milling was effectively used to produce nano-crystalline Al7075 alloy powders reinforced with 1, 3 and 5 vol.% Al2O3 at nano-size level (Mobasherpour, Tofigh & Ebrahimi 2013). XRD results indicated that the size of aluminum reached 32 nm after 20 hours of high-energy ball milling for Al/5Al2O3 nano-composite powder with uniform particle size distribution. Chaira, Sangal and Mishra (2007) employed specially built dual-drive planetary mill to first prepare Fe3C powder from iron and carbide powders and then mixed with Al powders to prepare Al/Fe3C composite powder. Further, composite powders of 10, 20 and 30 wt. % tungsten carbide and aluminum powder were produced in a ball mill machine for 2, 4 and 8 hours to investigate the mixing time on microstructural properties (Selte & Ozkal 2015).

**Step 4: Composite powder compaction in the required shape**

After the mixing and blending process, the required amount of powders are taken for the compaction process where high pressure is applied by the opposing punches squeezing powders contained in a die to form the powders into the required shape. The amount of powder to be compacted depends on the density and aspect ratio (height to diameter ratio) of the preform. Narayanasamy, Anandakrishnan & Pandey (2008) fabricated Al2O3-Al preforms with initial relative density of 0.35, 0.56 and 0.72 with compacting pressures of 93, 135 and 210 MPa, respectively, using a 0.60 MN hydraulic press was used with suitable die-set assembly containing die, a punch and a bottom insert. The die set assembly was made out of high carbon high chromium Steel. Molybdenum disulphide was used as a lubricant during compaction. Similar equipment and setup was used by Liu et al. (2006) to prepare titanium alloys and composites by cold isostatic pressing under a pressure of 200 MPa and Kumar, Narayanasamy and Loganathan (2012) to prepare green compacts with 0.92 relative density of Al–Glass–SiC hybrid composites by cold isostatic pressing under a pressure of 520 MPa.
Hot isostatic pressing can also be employed which eliminates a separate sintering process. Kai et al. (2011) stored and sealed the Ni–Cr–Co based super alloy powders in a stainless steel capsules which were subjected to hot isostatic pressing at 1180 °C for 4 hours at a pressure of 120 MPa. Similarly, Henriques et al. (2015) produce powder metallurgy CoCrMo alloy used in biomedical applications via hot isostatic pressing after the blending and mixing process. Knowles et al. (2014) produced 10 and 15 wt.% Al-SiC composites by a proprietary process including high energy ball milling followed by hot isostatic pressing. Sharifi and Karimzadeh (2011) first ball milled the respective powders to produce Al–(Al₂O₃–AlB₁₂) composite powders and then pressed at 450 °C and constant pressures of 300 MPa for 30 mins to produce Al–(Al₂O₃–AlB₁₂) composite material. In order to avoid pores development, the pressure was maintained until the specimen was cooled down.

Kumar, Narayanasamy and Loganathan (2011, 2012) followed the following procedure to remove the specimen from the die after the compaction process. The ejection of compact was carried out by taking out the bottom insert and placing the die on two parallel flat blocks keeping the die hole clear to have free space underneath the die hole and between the blocks. The compact is removed by applying the load on the punch and the compact came down in between the space provided by placing the supporting blocks. To aid this process the die, bottom insert and the punch are lubricated with zinc-stearate.

**Step 5: Ceramic coating of the green compacts**

A ceramic coating is generally applied after the compaction process before sintering to prevent oxidation of the compacts during the sintering process. The coating is generally applied twice with drying period of 10-12 hours after each coating. Reference (Narayanasamy, Senthilkumar & Pandey 2007) prepared an alumina mixed in acetone ceramic paste and coated the free surface of the preforms. This was left to dry for 6 hours in the atmospheric conditions. Then the second coating was applied in the direction 90° to that of the earlier coating and dried for further 12 hours in the atmospheric conditions. Similar process and technique was used by Kumar, Narayanasamy and Loganathan (2011, 2012) and Rajeshkannan and Sharma (2014). An alternative to this is sintering in a
controlled atmosphere with inert gas purging. Rajiv, Srinivasan and Balakrishnan (1997) prepared the green compacts and then sintered without any coating in an electrical muffle furnace in an inert atmosphere of dissolved ammonia for 2 hours.

*Step 6: Sintering of the ceramic coated green compacts*

After the compaction process via cold isostatic pressing, the compacts are called the green compacts as it is low in strength, however, enough to be handled during the sintering process. Sintering is the process whereby powder compacts are heated so that neighboring particles fuse together, thus resulting in a solid object with better mechanical strength compared to the green powder compact. Sintering temperatures used by many are around 0.8-0.9 melting temperature of the base material. Narayanasamy, Anandakrishnan and Pandey (2008) sintered ceramic coated Al-Al₂O₃ preforms in an electric muffle furnace in the range of 550 ± 10 °C for a period of 100 mins and ultimately cooled to room temperature in the furnace itself. Liu et al. (2006) sintered the titanium alloy and composite compacts at temperatures ranging from 1200 to 1350 °C for 3 hours in a vacuum of 5 × 10⁻³ Pa, followed by furnace cooling. Rajeshkannan and Sharma (2014) sintered ceramic coated hybrid composites of Al-2WC-4Fe₃C, Al-2WC-8Fe₃C and Al-2WC-12Fe₃C in an electric muffle furnace at 594 °C for 1 hour. Prior to the sintering process a drying process at 220 °C for 30 mins was carried out in the same furnace.

*Step 7: Cooling of the sintered preforms and secondary deformations as required*

Several cooling techniques are employed such as furnace cooling, air cooling, oil or water quenching and many more. Depending on the industrial application of the part, a secondary deformation process can be carried out to further increase the density and strength of the powder metallurgy parts. This secondary deformation process can be carried out at room temperature or at higher temperatures. Narayanasamy, Anandakrishnan and Pandey (2008) and Kumar, Narayanasamy and Loganathan (2012) carried out cold open die forging after furnace cooling of the sintered parts, whereas Rajeshkannan and Sharma (2014) carried hot open die forging immediately after the sintering process at the sintering temperature. Kai et al. (2011) reduced the specimen
height from 15 mm to 7.5 mm by hot compression tests at temperatures ranging from 950 °C to 1150 °C and immediately water cooled the specimens to freeze the microstructure. Liu et al. (2006) heated the sintered furnace cooled samples to 1100 °C and hot forged with a maximum strain of 50%. The deformed compacts were then held at 810 °C for 1 hour followed by water quenching and then annealing at 580 °C for 8 hours. Knowles et al. (2014) heated the samples to 450 °C for 20 mins and then extruded with an extrusion ratio of 14:1 producing 1 m long bars with 7.5 mm diameter.

2.4.2 Liquid stir casting

Stir casting technique also remains popular in the manufacture of aluminium metal matrix composites. Some common problems faced by stir casting processing technique are non-uniform distribution of the reinforcing particles in the matrix alloy, wettability, particle clustering, segregation, interfacial reaction and formation of detrimental secondary phases, however, well defined solution are reported to these problems (Boopathi, Arulshri & Iyandurai 2013; Umanath, Kumar & Selvamani 2013; Kumar and Dhimen 2013; Babu and Krishnan 2012). A schematic diagram of the stir casting setup is shown in Fig. 2.1 and the processing steps are discussed below.

![Figure 2.1: Layout of stir casting setup.](image)
Step 1: Selection of matrix and reinforcement materials
Aluminium alloy of any size and type may be chosen to prepare the composite. SiC and Al₂O₃ are commonly used as reinforcing particles in the production of aluminium metal matrix composites, however, other common reinforcements used recently are titanium carbide, tungsten carbide, boron carbide, boron and graphite. The particle sizes of these reinforcements generally range from 30-60 \( \mu m \).

Step 2: Selection of volume fraction of reinforcement materials
This can be selected depending on specific applications as the presence of the reinforcement produces changes in the microstructure of the matrix and therefore to the overall properties.

Step 3: Melting of matrix alloy
The matrix alloy is cleaned properly before subjected to the melting process in a temperature range of 700 °C to 950 °C. Some researchers preheated the matrix just before the melting process to make its surface oxidized. Mamatha, Pruthviraj and Ashok (2011) used distilled water, Babu and Krishnan (2012) used acetone and Kayal et al. (2011) used distilled water to wash the aluminium matrix alloy. Other researchers chose not to wash the matrix alloy and went directly to the preheat process (Sahin 2003; Safiuddin et al. 2010; Henriques et al. 2015). Sahin (2003) preheated the aluminium powder at 200 °C for 1 hour before increasing the temperature to 750 °C for melting. Similarly, Kayal et al. (2011) conducted preheating at 500 °C and melting at 760 °C. On the other hand, Alaneme and Olubambi (2013), Srikanth and Amarnath (2015) and Shehata et al. (2013) directly melted the matrix alloy at 750 °C, 850 °C and 750 °C, respectively. Mamatha, Pruthviraj and Ashok (2011) used nitrogen gas, whereas Kok (2005) and Cocen and Onel (2002) used argon gas as a protective layer over the melt during the melting process. On the other hand, some researchers did not use any protective gas layering during the melting process (Sahin 2003; Alaneme and Olubambi 2013; Kayal et al. 2011). Finally, the molten metal needs to be degasified by some suitable degasser, like solid dry hexachloroethane (Srikanth & Amarnath 2015).
Step 4: Addition of preheated reinforcement into the molten matrix alloy

The reinforcement particles are preheated to certain temperature before addition to the melted matrix alloy. This is done to remove moisture and increase the wettability of the particles. Sahin (2003) preheated SiC to 1100 °C for 2 hours, Murthy and Singh (2015) preheated TiC to 400 °C and Srikanth and Amarnath (2015) preheated WC to 500 °C before introducing it into the molten matrix alloy.

Next the melt aluminium matrix alloy is stirred and preheated reinforcement is introduced in the melt with the help of the vortex created by stirring. Mamatha, Pruthviraj and Ashok (2011) stirred the molten aluminium at the speed of 450 rpm and preheated SiC is added at 120 g/min. Murthy and Singh (2015) used a stirring speed of 450 rpm and preheated TiC is added at 20 g/min. Srikanth and Amarnath (2015) used a stirring speed of 500 rpm and preheated WC were added slowly into the melt.

Alaneme, Tolulope and Peter (2014) preferred stirring the aluminium matrix alloy in semi-solid state at 600 °C (temperature just below the melting point) at 400 rpm and added preheated SiC into the melt with the aid of the vortex created. Naher, Brabazon and Looney (2004) also added the preheated SiC in the semi-solid state with stirring speed ranging from 200-500 rpm. The mixing time taken is generally in a range of 10-20 mins. To avoid contamination stirrers are usually coated with alumina.

In many cases a wetting agent, like pure magnesium powder or borax is added into the molten composite slurry after the reinforcements in a similar way. The proportion is generally in a range of 1-3 wt.%. this is added to achieve a good bond between the reinforcement and matrix alloy (Alaneme & Olubambi 2013).

Step 5: Pouring of the molten composite slurry into a preheated mold

After sufficient mixing for about 10-20 mins, the pouring of the molten composite slurry in the temperature range of 680 °C to 750 °C to the preheated mold is carried out. The mold may be preheated to the temperature range of 250 °C to 760 °C (Kok 2005; Mamatha, Pruthviraj & Ashok 2011; Alaneme & Olubambi 2013).
Finally, the mold is left to solidify at different cooling rates, atmospheric cooling, water quenching and solution treatment at different temperatures (Alaneme, Tolulope and Peter 2014; Murthy & Singh 2015; Naher, Brabazon & Looney 2004).

After extensive literature review, it is noticed that both these processing techniques are excellent in the production of aluminium metal matrix composites. This section highlights the different combination of reinforcements used in the production of aluminium metal matrix composites. It is still difficult to justify the performance amongst single reinforced, hybrid reinforced and hybrid ceramic/agro-waste reinforced aluminium metal matrix composites. More studies are required to optimize reinforcements and their volume fraction together with the production process to determine the optimum processing parameters. An attempt has also been made to outline the fabrication methods of aluminium metal matrix composites, giving special attention to powder metallurgy and stir casting techniques. So far it was seen that these two techniques are well documented and used successfully in the production of aluminium metal matrix composites.

2.5 Importance of densification, workability, forming limit and corrosion studies on the fabrication of powder metallurgy parts.

2.5.1 Densification behavior

Densification behavior is an important study in the preparation and performance of powder metallurgy materials. The behavior is very different and not easy to predict when more than one element is present in the powder material. Further, it is difficult to predict the densification behavior when the hardness values vary significantly (Tiwari, Rajput & Srivastava 2012). Fabian and Selvam (2014) studied the densification behavior of WC/Al aluminium composites prepared by powder metallurgy method. To expose the densification behavior they studied the final density achieved with varying wt. % of WC reinforcements in the matrix, final density achieved with varying geometry and final density achieved after the sintering process. Padmavathi and Upadhyaya (2010) studied the influence of SiC reinforcement on densification and mechanical properties of supersolidus liquid phase sintered 6711Al-SiC composites. They reported that the densification response of unreinforced aluminum alloys was improved when sintered in
the presence of liquid phase. Further, a uniform distribution of the reinforcement particles in the matrix alloy was seen up to 10 vol.% SiC resulting in better density, yield strength, wear and corrosion resistance. More than 10 vol. % SiC resulted in clustering of SiC particles and had negative effect on densification and mechanical properties.

2.5.2 Workability behavior

Workability studies provide the ease with which a material can be shaped through plastic deformation without failure (Hassani, Bagherpour & Qods 2014). A lot of parameters such as mechanical alloying, microstructure, sintering temperature, reinforcement materials and strain rate affect the workability behavior of powder metallurgy materials (Shao et al. 2010; Hassani, Bagherpour & Qods 2014). Plastic deformation of powder metallurgy materials compared to conventional materials is complicated due to the presence of porosity. For this reason powder metallurgy materials experience volume changes and the mode of deformation depends on the density as well as on the hydrostatic stress (Rahman & El-Sheikh 1995). Taleghani and Torralba (2013) studied hot deformation behavior and workability characteristics of magnesium alloy powder compacts to expose the optimum hot working window. Raj et al. (2012) presented the effect of sintering time and sintering temperature on the workability characteristics of SiC reinforced aluminium composites. Some researchers studied the effect of volume fraction of reinforcement in the matrix composite, initial geometry and initial relative density on the workability behavior of powder metallurgy materials (Das et al. 2010; Narayanasamy, Ramesh & Pandey 2005; Narayan & Rajeshkannan 2013). Several yield criterions are used to study the workability behavior of the powder metallurgy materials. Shao et al. (2010) used modified Arrhenius-type constitutive equations to study the constitutive flow behavior and hot workability of powder metallurgy materials. Further, they used modified dynamic material model to construct the powder dissipation efficiency maps and Ziegler’s and Gegel’s stability criterion to construct instability maps. Narayanasamy et al. (2008) studied the workability behavior of the powder metallurgy materials using the generalized yield criterion developed under triaxial stress state. Same yield criterion was used by several other researchers (Hassani, Bagherpour & Qods 2014; Raj et al. 2013; Rajeshkannan 2010).
2.5.3 Forming limit study

The occurrence of ductile fracture during the plastic deformation of powder metallurgy materials is adverse and damaging and the prediction of fracture is very important in the early stages as early modifications will prevent failure. This will tend to save a lot of money and forming limit studies in many metal forming processes is up most important. Poshal and Ganesan (2008) predicted the values of deformation characteristics and formability factor using a proposed network model. They tested the model with experimental values and concluded that it is a promising technique and capable of predicting forming limit parameters together with production advice relating to many aspects of powder metallurgy including design, production and control. In view of this, forming limit analysis of powder metallurgy materials were carried out by many researchers. (Butuc, Gracio & Rocha 2006; Selvakumar et al. 2013; Huang & Cheng 2005).

2.5.4 Corrosion studies

It is expected that the corrosion resistance of powder metallurgy materials will degrade with increasing number of interfaces. However, this is still under investigations as many other features also influence the corrosion behavior of powder metallurgy materials such as the production route, the heat treatment, the surface treatment, the secondary phase nature and content (Lekatou et al. 2015). A corrosion and wear analysis was carried out by Lekatou et al. (2015) to understand the performance of WC and TiC reinforced aluminium matrix composites prepared by stir casting route. Cyclic potentiodynamic polarization technique was used in Dilute Harrison’s Solution. It was seen that the corrosion behavior of the composites was mainly affected by the corrosion of the alloy matrix rather than the reinforcement/matrix interface. Similarly, Griffiths and Turnbull (1994) showed that the corrosion did not increase much due to the reinforcing particle but by the alloy matrix. On the other hand, Pardo et al. (2005) presented that the corrosion destruction in AA360/SiC and AA380/SiC composites was initiated by pitting attack largely at the reinforcement/matrix interface. Similarly, Zuhair and Amro (2002) and Sherif et al. (2011) noted that the corrosion behavior was accelerated at the reinforcement/matrix interface and reinforcement clusters rather than at the matrix alloy.
3.1 Experimental details

3.1.1 Materials and characterization

Aluminium powder of less than or equal to 150 µm in size (diameter) and respective carbide powders, namely, titanium carbide, tungsten carbide, molybdenum carbide and iron carbide, of less than or equal to 50 µm in size (diameter) were used in this experiment. The basic characterization of elemental aluminium powder such as flow rate, apparent density, compressibility, and sieve analysis has been carried out using standard methods of testing. All powders used in the present investigation had purity levels of 99.7%. The characteristics are given in Tables 3.1 and 3.2, respectively. A sieve shaker shown in Fig. 3.1 constructed in the Mechanical Laboratory of The University of the South Pacific is used for the sieving activities. A hall flow meter (Fig. 3.2) is used to measure the flow rate.

![Sieve shaker](image)

Figure 3.1: Sieve shaker

<table>
<thead>
<tr>
<th>Sieve size (µm)</th>
<th>250</th>
<th>+200</th>
<th>+150</th>
<th>+100</th>
<th>+75</th>
<th>+45</th>
<th>-45</th>
</tr>
</thead>
<tbody>
<tr>
<td>Retention in sieve (Weight %)</td>
<td>0.2</td>
<td>0.3</td>
<td>16.3</td>
<td>55.3</td>
<td>9.5</td>
<td>7.9</td>
<td>10.5</td>
</tr>
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</table>
### Table 3.2: Characterization of aluminium powder and its blends

<table>
<thead>
<tr>
<th>Property</th>
<th>Al</th>
<th>Al-1TiC</th>
<th>Al-2TiC</th>
<th>Al-3TiC</th>
<th>Al-4TiC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apparent Density (g/cc)</td>
<td>1.091</td>
<td>1.133</td>
<td>1.186</td>
<td>1.247</td>
<td>1.186</td>
</tr>
<tr>
<td>Flow rate, (s/50g) by Hall Flow Meter</td>
<td>87.306</td>
<td>86.801</td>
<td>85.202</td>
<td>83.086</td>
<td>85.202</td>
</tr>
<tr>
<td>Compressibility (g/cc) at pressure of 130±10MPa</td>
<td>2.356</td>
<td>2.342</td>
<td>2.325</td>
<td>2.302</td>
<td>2.280</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Property</th>
<th>Al-2WC</th>
<th>Al-2Fe₃C</th>
<th>Al-2Mo₂C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apparent Density (g/cc)</td>
<td>1.262</td>
<td>1.236</td>
<td>1.258</td>
</tr>
<tr>
<td>Flow rate, (s/50g) by Hall Flow Meter</td>
<td>78.848</td>
<td>78.909</td>
<td>78.132</td>
</tr>
<tr>
<td>Compressibility (g/cc) at pressure of 130±10MPa</td>
<td>2.273</td>
<td>2.313</td>
<td>2.301</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Property</th>
<th>Al-4WC</th>
<th>Al-4Fe₃C</th>
<th>Al-4Mo₂C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Apparent Density (g/cc)</td>
<td>1.345</td>
<td>1.308</td>
<td>1.325</td>
</tr>
<tr>
<td>Flow rate, (s/50g) by Hall Flow Meter</td>
<td>79.647</td>
<td>80.559</td>
<td>80.481</td>
</tr>
<tr>
<td>Compressibility (g/cc) at pressure of 130±10MPa</td>
<td>2.113</td>
<td>2.235</td>
<td>2.210</td>
</tr>
</tbody>
</table>
3.1.2 Selection of given relative densities and aspect ratio

Generally, it is seen that the initial relative density also affects many properties of powder metallurgy materials and the safe working initial density are generally above 80% of theoretical density (Narayanasamy, Ramesh & Pandey 2005; Narayan & Rajeshkannan 2011; Shanmugasundaram & Chandramouli 2009). For ease of manufacturing and load requirements the upper limit are mostly 90% of theoretical density. Hence, two initial relative densities of 0.82 and 0.86 are chosen to see the effect of initial relative density on the densification behavior.

The aspect ratio (height to diameter ratio) of 0.2, 0.4 and 0.6 are chosen to understand the effect of geometry on the densification behavior. By changing the aspect ratio we have varied the number of pores in the manufactured part. Further, the pore bed height is also varied with varying aspect ratio and the effect of this is investigated. This aspect ratio was chosen based on the average sizes of powder metallurgy industrial parts.

3.1.3 Powder blending and compaction

The required mass of aluminium and respective carbide powders were accurately weighed and mixed to obtain Al-1TiC, Al-2TiC, Al-3TiC, Al-4TiC, Al-1Fe3C, Al-2Fe3C,
Al-4Fe3C, Al-6Fe3C, Al-1Mo2C, Al-2Mo2C, Al-3Mo2C, Al-4Mo2C, Al-1WC, Al-2WC, Al-3WC and Al-4WC in a planetary ball milling machine, model Retsch PM400 (Fig. 3.3). No mixing balls were employed in the mixing process throughout the research work as the required size of powders was available. Air tight stainless steel containers were used here to avoid oxidation of aluminium powders. These were supplied with the machine. Argon purging was not required. The ball mill was operated for 2-2.5 hours at 200 rpm to get a homogenized mixture. The apparent density was measured continuously to ensure homogenous mixture was obtained. Towards the end of blending process a consistent apparent density ensured homogeneous mix. The blended powders were then compacted using 100 tons capacity hydraulic press (Fig. 3.4) into cylindrical billets of aspect ratio (height-to-diameter ratio) of 0.2, 0.4 and 0.6. The respective compacting pressures were obtained from the compressibility curve (Fig. 3.6) prepared for each material so as to obtain an initial theoretical density of 0.82 ± 0.01 and 0.86 ± 0.01. On average the compacting pressure requirements were in a range of 139MPa to 159MPa. Initial theoretical density is initial density of the preform divided by 100% density of the preform (no pores).
Figure 3.4: 1.0 MN hydraulic press.

Figure 3.5: Die-set assembly & upsetting dies.
3.1.4 Ceramic coating, drying and sintering

Immediately after compaction an indigenously developed ceramic coating, Al₂O₃ mixed with acetone, was applied on the compacts. This coating was allowed to dry for a period
12 hours at normal atmospheric conditions. Recoating was employed to the preforms in the direction 90° to that of the earlier coating. Again the compacts were allowed to dry for a period of 12 hours. The coating was applied to avoid oxidation of compacts during the sintering process. The ceramic coated compacts were sintered in an electric muffle furnace (Fig. 3.7) at a temperature of 220°C for 30 minutes (drying process) and then at the temperature of 594°C for further 60 minutes.

3.1.5 Hot deformation and measurements

Eight specimens of each material and aspect ratio was prepared and sintered. Hot upsetting of the seven sintered preforms followed immediately after the sintering process at a temperature of 594°C to the different levels of height strains until visible cracks can be seen on the free surface. All seven deformed specimens and one un-deformed specimen was left in the open air for atmospheric cooling. It was ensured that one of the specimens had visible crack on the bulging surface while others were prevented from this defect. The hot specimens directly from the furnace were placed between two flat dies and the impact load (varied to achieve different height strains) was provided by the dropping hammer. The height of the dropping hammer was varied to achieve different levels of height strain. Only the gravitational force of the dropping hammer was employed in the deformation process. The deformation was achieved in one or two blows of the hammer. Where two blows were required on the same specimens, the specimen was turned up-side down after the first blow and again placed between the two flat dies. Then the second blow was taken. The whole process was done in quick succession to avoid the specimen from cooling down. The forging operation was carried out with no lubricant. The effect of lubrication was not studied in this work as the authors concentrated on other aspects such as geometry, initial relative density, and mainly the composites. Further, the risk involved using a lubricant in hot forging such as pollution, smoke emission, corrosive, etc. was high as the authors were involved in the production of high number of specimens. Finally, the compacts were left for atmospheric cooling and then the geometrical and density (initial and deformed) measurements were taken. Dimensional measurements such as deformed height ($h_d$), deformed diameters, namely, contact diameter at the top surface ($D_{c1}$), contact diameter at the bottom surface ($D_{c2}$),
bulged diameter \((D_b)\), were carried out using digital Vernier caliper after every step of deformation. The density measurements of the forged specimens were carried out using Archimedes principle with 0.1 gram accuracy weighing balance as shown in Fig. 3.8. Experimental data’s were used to calculate the stress ratio parameters, namely, \((\sigma_\theta / \sigma_{\text{eff}})\), \((\sigma_m / \sigma_{\text{eff}})\) and \((\sigma_z / \sigma_{\text{eff}})\), axial strain, hoop strain, effective strain, true diameter strain, percent relative density, actual bulged length, axial stress, effective stress, hoop stress, hydrostatic stress, formability stress index, instantaneous strength coefficient \((K_i)\) and instantaneous strain hardening exponent \((n_i)\).

The microstructure was also taken for the final deformed preform at the center and at the diametrical side. The deformed preform was cut into two halves diametrically and the specimen was prepared for the microstructure viewing in Olympus microscope with model number PMG 3. A 3-CCD high resolution video camera, model number KY-F55BE, was used to capture the microstructure as shown in Fig. 3.9.

![Figure 3.8: Kern weighing balance to 0.1g accuracy.](image)
Figure 3.9: Olympus microscope with 3-CCD high resolution video camera.
Chapter 4

Densification behavior of sintered aluminum composites during hot deformation

4.1 Overview

Densification behavior plays a key part in the enhancement of the aforementioned properties in the aluminium metal matrix composites. The static mechanical properties are directly related and affected by the residual porosity, however, the dynamic mechanical properties depend on both the residual porosity and the mode by which full densification is achieved. Densification is greatly affected by the stress state in the material and the stress state during deformation is not uniform throughout the workpiece and hence densification is not uniform. The pores present in the powder metallurgy materials especially the open ones has significant effect on the densification behavior and solving these problems will increase the potential use of powder metallurgy parts for industrial applications. Chandramouli et al. (2007) presented densification behavior of plain carbon steels (Fe and Fe-1%C) and its relevance on the corrosion and deformation behavior. They presented that the rate of densification was higher for 1%C with Fe during both cold and hot upsetting. The maximum density achieved in both materials was 97% theoretical density regardless of the deformation method. Lower densified preforms showed greater rate of uniform corrosion in both Fe and Fe-1%C carbon steels. Danninger, Gierl and Salak (2009) have reported the correlation between material density, Vickers hardness and tensile strength in sintered powder metallurgy iron and steels specimens. It was reported that the final density achieved by the specimen had great effect on the hardness and tensile properties. Further, it was reported (Danninger et al. 1993a, 1993b) that the mechanical properties such as impact, tensile and fatigue properties of powder metallurgy steels are greatly influenced by the relative density, sintering time and temperature. Hence, densification studies are absolute necessary and the necessary changes in the processing techniques can be implemented in the production of such structural parts.
Rahimian et al. (2009) studied the influence of sintering time and temperature and particle size on the densification behavior as well as other properties. They showed that the particle size and sintering time and temperature has different densification properties and this also affects other properties such as hardness, yield strength, compressive strength and elongation to fracture.

Powder preform forging is a secondary deformation method employed to conventionally make powder metallurgy parts to achieve the near net final shape enhancing density of the product simultaneously for structural and heavy duty applications. Rajeshkannan et al. (2008) performed experimental studies to analyze deformation and densification behavior sintered high carbon alloy powder metallurgy steel. It was reported that in the process of increasing density of the powder metallurgy materials, the strength and strain also increased; however, the work hardening behavior was not homogeneously increased with strain and densification.

The material yield stress plays an important role in achieving the compacted part’s final strength and density. A new deformation model was presented (Al-Qureshi, Galiotto & Klein 2005; Al-Qureshi et al. 2008) which included modifying Mohr-Coulomb and work hardening hypothesis which included internal friction, die-wall friction and work hardening during the compaction process. Other factors that also affected the final compacted density were starting density of the compact and the pressure distribution from the center to the edge of the preform for a given friction condition, lubricated powders and higher particle size.

Zhou et al. (2002) presented a densification mathematical model for porous metallic powder materials. They reported that the plastic deformation of the metallic material is mostly caused by the slipping of crystal lattice. This principle can be used effectively to improve the density and strength in the powder metallurgy materials. Further, they introduced voids in the FEM model making it possible to carry out simulations correctly and in a timely manner.
Hua et al. (2006) presented and validated several yield criterion that are used to study plastic deformation behavior of compressible powder metallurgy materials. The proposed yield criterion can predict the initial yield as well as successive yielding point of compressible sintered powder metallurgy materials. The effects of initial density and densification strengthening are depicted by the proposed criteria.

4.2 Theoretical analysis

Using the mathematical expressions the various upsetting parameters that influence workability characteristics of the selected composite were determined and presented here. The state of stress in a homogeneous compression process is as follows: According to Rahman and El-Sheikh (1995):

\[
\sigma_z = \frac{\text{load}}{\text{contact surface area}} = -\sigma_{eff}, \quad \sigma_r = \sigma_\theta = 0
\] (4.1)

\[
\sigma_m = \left( \frac{\sigma_z}{3} \right) = -\left( \frac{\sigma_{eff}}{3} \right)
\] (4.2)

and the expression for the axial strain can be written as follows:

\[
\varepsilon_z = -\varepsilon_{eff} = \ln \left( \frac{h_f}{h_o} \right)
\] (4.3)

and true hoop strain is

\[
\varepsilon_\theta = \varepsilon_r = \ln \left( \frac{D_f}{D_o} \right)
\] (4.4)

where \(h_o\) is the initial height of the preform; \(h_f\) the forged height of the preform; \(D_f\) the contact diameter after deformation of the preform; \(D_o\) the initial diameter of the preform.

According to Raj et al. (2013), Ramesh, Prabhakar & Narayanasamy (2009) and Narayanasamy, Ramesh & Pandey (2005, 2006), the hoop strain under plane stress state which includes the forged bulged diameter \((D_b)\) and forged contact diameter \((D_c)\) can be expressed as follows

\[
\varepsilon_\theta = \ln \left[ \frac{2D_b^2 + D_c^2}{3D_o^2} \right]
\] (4.5)

Plastic deformation of powder metallurgy materials is effected by the residual pores and the analysis of such materials requires an appropriate yield criterion which should take
the pore effect into account. Many researchers over the years have analyzed several different yield criteria for sintered powder materials and a typical theorem is that the plastic deformation occurs when the elasticity strain energy reaches a critical value (Qin & Hua 2007; Han, Oh & Lee 1995; Lewis & Khoei 2001). The formulation can be written as

\[ AJ'_2 + BJ'_1 = Y^2 = \delta Y_0^2 \]  

(4.6)

where \( A, B, \delta \) are yield criterion parameters and are functions of relative density, \( J_1 \) is the first invariant of the stress tensor, \( J'_2 \) is the second invariant of the stress deviator and \( Y_0 \) and \( Y \) are yield strength of a solid and partially dense material having relative density \( R \), respectively (Lewis & Khoei 2001). The parameters \( J_1 \) and \( J'_2 \) in the cylindrical coordinate system where the axis represents radial, circular and axial direction can be expressed as follows

\[ J'_2 = \frac{1}{6} \left[ (\sigma_r - \sigma_\theta)^2 + (\sigma_\theta - \sigma_z)^2 + (\sigma_z - \sigma_r)^2 \right] \]  

(4.7)

\[ J_1 = \sigma_r + \sigma_\theta + \sigma_z \]  

(4.8)

Here for axisymmetric forging, \( \sigma_r = \sigma_\theta \), \( J'_2 \) and \( J'_1 \) can be written as

\[ J'_2 = \frac{1}{6} \left( 2\sigma_\theta^2 + 2\sigma_z^2 - 4\sigma_\theta \sigma_z \right) \]  

(4.9)

\[ J'_1 = 4\sigma_\theta^2 + \sigma_z^2 + 4\sigma_\theta \sigma_z \]  

(4.10)

Substituting Eq. (4.9) and Eq. (4.10) into Eq. (4.6) gives

\[ A \left( 2\sigma_\theta^2 + 2\sigma_z^2 - 4\sigma_\theta \sigma_z \right) + B \left( 4\sigma_\theta^2 + \sigma_z^2 + 4\sigma_\theta \sigma_z \right) = \delta Y_0^2 \]  

(4.11)

Qin and Hua (2007) have investigated and compared several yield criterion parameters based on plastic Poisson’s ratio, relative density and flow stress of the matrix material by previous researchers. The following yield criteria parameters are chosen in this research as \( A = 2 + R^2 \), \( B = (1 - R^2) / 3 \), \( \delta = 2 R^2 - 1 \). Eq. (4.11) can now be written as

\[ Y_0 = \sigma_{eff} = \left[ \frac{(\sigma_z^2 + 2\sigma_\theta^2 - R^2(\sigma_\theta^2 + 2\sigma_\theta \sigma_z))}{2R^2 - 1} \right]^{0.5} \]  

(4.12)
Eq. (4.12) gives the expression for effective stress in terms of cylindrical coordinates and can be expressed in another form as

\[
\frac{\sigma_{\text{eff}}}{\sigma_z} = \left[1 + 2\left(\frac{\sigma_\theta}{\sigma_z}\right)^2 - \frac{R^2(2(\sigma_\theta/\sigma_z) + (\sigma_\theta/\sigma_z)^2)}{2R^2 - 1}\right]^{0.5}
\]  

(4.13)

According to Narayanasamy, Ponlagusamy & Subramanian (2001), the state of stress in a triaxial stress condition is given by

\[
\alpha = \frac{d\varepsilon_\theta}{d\varepsilon_z} = \frac{(2 + R^2)\sigma_\theta - R^2(\sigma_z + 2\sigma_\theta)}{(2 + R^2)\sigma_z - R^2(\sigma_z + 2\sigma_\theta)}
\]  

(4.14)

Using Eq. (4.14) for the values of Poisson’s ratio (\(\alpha\)), relative density (\(R\)) and axial stress (\(\sigma_z\)) the hoop stress (\(\sigma_\theta\)) under triaxial stress state condition can be determined as given below:

\[
\frac{d\varepsilon_\theta}{d\varepsilon_z}[(2 + R^2)\sigma_z - R^2(\sigma_z + 2\sigma_\theta)] = (2 + R^2)\sigma_\theta - R^2(\sigma_z + 2\sigma_\theta)
\]  

(4.15)

\[
\sigma_\theta = \left[\frac{2\alpha + R^2}{2 - R^2 + 2R^2\alpha}\right]\sigma_z
\]  

(4.16)

where, \(\alpha = \frac{d\varepsilon_\theta}{d\varepsilon_z}\)

Further, rearranging Equation 4.16

\[
\frac{\sigma_\theta}{\sigma_z} = \left[\frac{2\alpha + R^2}{2 - R^2 + 2R^2\alpha}\right]
\]  

(4.17)

Under triaxial stress state cylindrical coordinates, the hydrostatic stress can be written as follows assuming \(\sigma_r = \sigma_\theta\):

\[
\sigma_m = \frac{\sigma_r + \sigma_\theta + \sigma_z}{3} = \frac{2\sigma_\theta + \sigma_z}{3}
\]  

(4.18)

Further, rearranging Equation 4.18

\[
\frac{\sigma_m}{\sigma_z} = \frac{1}{3}\left(1 + \frac{2\sigma_\theta}{\sigma_z}\right)
\]  

(4.19)
The formability stress index ($\beta$) is used to describe the effect of hydrostatic stress ($\sigma_m$) and the effective stress ($\sigma_{eff}$) on the forming limit of powder metallurgy materials during upset forging. The stress formability factor under triaxial stress state condition is given as

$$\beta = \frac{3}{1} \left[ \frac{\sigma_m / \sigma_z}{\sigma_{eff} / \sigma_z} \right]$$ (4.20)

The stress formability factor as expressed in Eq. (4.20) is used to describe the effect of mean stress and the effective stress on the forming limit of powder metallurgy compacts in upsetting.

Different stress ratio parameters, namely, ($\sigma_\theta / \sigma_{eff}$), ($\sigma_m / \sigma_{eff}$) and ($\sigma_z / \sigma_{eff}$) are expressed as follows:

$$\frac{\sigma_\theta}{\sigma_{eff}} = \frac{\sigma_\theta / \sigma_z}{\sigma_{eff} / \sigma_z}$$ (4.21)

$$\frac{\sigma_m}{\sigma_{eff}} = \frac{\sigma_m / \sigma_z}{\sigma_{eff} / \sigma_z}$$ (4.22)

$$\frac{\sigma_z}{\sigma_{eff}} = \frac{1}{\sigma_{eff} / \sigma_z}$$ (4.23)

4.3 Results and discussions

The hard carbide particles which is present in aluminium composite tested here will have some effect on densification. Since some of the properties of the hard carbide particles and its blends as seen in Table 4.1 are different, the densification and deformation behavior would be different as well as the final density attained would be different. A plot has been presented as shown in Fig. 4.1 between axial strain and relative density for two different initial aspect ratios (height-to-diameter ratio of preforms), namely, 0.40 and 0.60, these plots being drawn for initial relative density of 86%. Similarly Fig. 4.2 is drawn for initial relative density of 82%. Relative density is the instantaneous density of the preform divided by 100% density of the preform (no pores).
As seen in Fig. 4.1 the relative density increases up to 0.45 axial strain and thereafter relative density is almost constant. The densification rate is higher at lower height strain, however, this effect is found to be different in various materials tested in this study. This is because of the larger and higher amount of pores present during the initial stages of deformation which closes with small axial strains. Rudenko & Laptev (2011) also presented similar behavior while studying aluminium metal matrix composite via cold deformation. As the deformation progresses the material increases in strength as a result of reduction of pores thereby increasing in relative density. The pore closure rate is directly proportional to densification; hence, even though the height strain is prominent at the final stage, the densification rate is low. The main reason here is that the cylindrical pores elongate in the lateral direction with the flow of material which is perpendicular to the direction of load application. As the deformation progresses the relative density, grain boundary growth and the strength of the material improves. To further improve the relative density by eliminating more pores is difficult as the mobility of the particles decrease due to the improvement in relative density, strength and grain boundary growth for every stage of deformation. Thus the densification rate reduces significantly during the final stages of deformation. Al-4TiC showed better densification at final stages of deformation. Similar findings and outcomes were presented by Narayan and Rajeshkannan (2011) and Rajeshkannan and Narayan (2009). TiC reinforced aluminium showed better densification rate and better final density achieved followed by Fe₃C reinforced aluminium and then Mo₂C reinforced aluminium. WC reinforced aluminium had the lowest densification rate and final density achieved. However, an inverse relationship exists between densification and fracture strain for the respective composites. As seen in Table 4.1, the amount of TiC powder required is more to form Al-4TiC when compared to the amount of WC powder required to form Al-4WC composite. Further, aluminium particles are bigger in size (150 μm) in comparison to carbide particles (50 μm). This means that the powder stacking will be better in Al-4TiC compared to other composite. Also the percentage of pores is same in all the composites; however, smaller pores are present in Al-4TiC composite. These will aid in densification process, hence, better densification is observed for TiC containing composites. Shear modulus plays an important role in plastic deformation of metals. Shear modulus in metals usually ranges
from 20000 to 150000 MPa, however, the shear stress required for plastic deformation are always lower when compared with shear modulus values of that material. As the shear modulus are usually high in metals it is difficult to balance with smaller shear stress required for plastic deformation. Further, shear modulus is directly proportional to shear strength \( T_m = G/2\pi; \) where \( T_m \) is the shear strength and \( G \) is the shear modulus). From Table 4.1 it can be seen that the shear modulus of WC is much greater than shear modulus of TiC, hence the density is lower in Al-4WC when compared to Al-4TiC. In many materials, dislocation is the carrier of plastic deformation and the energy required to move them is less than the energy required to fracture the material. Lower aspect ratio preforms showed better densification in comparison to higher aspect ratio due to the presence of lower pore bed height in the lower aspect ratio preforms. Further, for Al-4TiC smaller aspect ratio preform also had the higher final density attainment, however, for all other composites the final density attainment is almost same irrespective of aspect ratio. The number of pores present in the higher aspect ratio preform is more than the number of pores present on the smaller aspect ratio preform. Hence, the densification rate is higher in smaller aspect ratio preform. Further, when the initial relative density is lower the final density achieved by all the composites are lower compared to higher initial relative density preforms (Figures 4.1 and 4.2). More number of pores is present in lower initial relative density preform and the chances of crack initiation will be more in lower initial relative density preform compared to higher relative density preform. This could be the cause for low final density attainment by lower initial relative density preform as chances of crack initiation before good final density attainment is highly possible. Similar finding are presented by Narayanasamy, Senthilkumar and Pandey (2007).

Further, a plot has been presented as shown in Fig. 4.3 between diametrical strain and axial strain for two different initial aspect ratios, namely, 0.40 and 0.60, these plots being drawn for initial relative density of 86%. As seen in Fig. 4.3 the lateral deformation is highest in WC reinforced aluminium composite followed by Mo\(_2\)C, then Fe\(_3\)C and then TiC composites. This means the effective closer of pores is higher in TiC reinforced aluminium, therefore, it has higher density than any other composite tested.
Table 4.2 shows the equations obtained using polynomial best fit graphs with correlation values close to 1.0 for relative density, $R$, vs axial strain, $\varepsilon_z$. It can be seen that for zero height strain a constant value of approximately 0.86 is obtained. This constant will change accordingly if the initial relative density is varied as the constant represents the initial relative density. Further, the first order value in Table 4.2 is found to be positive meaning it is contributing to the densification linearly. Also it is seen that the $\varepsilon_z$ coefficient increases as the aspect ratio decreases, projecting that decreasing aspect ratio promotes densification. The second order coefficient is found to be negative and hence its contribution is negative to densification and this negative contribution is more in lower aspect ratio preforms. However, the effect is less as the coefficient values are small in a range of 0-0.3 and is multiplied to the square of axial strain.

Table 4.1: Properties of respective powders and its blend

<table>
<thead>
<tr>
<th>Powder/Blend</th>
<th>Al</th>
<th>TiC</th>
<th>Fe$_3$C</th>
<th>Mo$_2$C</th>
<th>WC</th>
<th>Al-4TiC</th>
<th>Al-4Fe$_3$C</th>
<th>Al-4Mo$_2$C</th>
<th>Al-4WC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (g/cc)</td>
<td>2.70</td>
<td>4.90</td>
<td>7.70</td>
<td>8.90</td>
<td>15.80</td>
<td>2.79</td>
<td>2.90</td>
<td>2.95</td>
<td>3.23</td>
</tr>
<tr>
<td>Shear modulus (GPa)</td>
<td>-</td>
<td>188</td>
<td>-</td>
<td>-</td>
<td>270</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Apparent density (g/cc)</td>
<td>1.091</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1.186</td>
<td>1.308</td>
<td>1.325</td>
<td>1.345</td>
</tr>
<tr>
<td>To prepare 200g of Al4xx</td>
<td>Mass (g)</td>
<td>192</td>
<td>8</td>
<td>8</td>
<td>8</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Volume (cm$^3$)</td>
<td>71.11</td>
<td>1.63</td>
<td>1.04</td>
<td>0.90</td>
<td>0.51</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
Figure 4.1: Relationship between relative density and axial strain during hot deformation for initial relative density of 0.86.

Figure 4.2: Correlation of relative density and axial strain for initial relative density of 0.82.
Figure 4.3: The variation of the diameter strain with respect to the axial strain during hot deformation for initial relative density of 0.86.

Table 4.2: Polynomial curve fitting result – $R$ vs $\varepsilon_z$ for initial relative density of 0.86.

<table>
<thead>
<tr>
<th>Material</th>
<th>$R_0$</th>
<th>$R$ vs $\varepsilon_z$</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-4TiC</td>
<td>0.4</td>
<td>$R = -0.2922\varepsilon_z^2 + 0.2834\varepsilon_z + 0.8650$</td>
<td>0.9958</td>
</tr>
<tr>
<td></td>
<td>0.6</td>
<td>$R = -0.2395\varepsilon_z^2 + 0.2579\varepsilon_z + 0.8578$</td>
<td>0.9905</td>
</tr>
<tr>
<td>Al-4Mo$_2$C</td>
<td>0.4</td>
<td>$R = -0.1966\varepsilon_z^2 + 0.1725\varepsilon_z + 0.8602$</td>
<td>0.9970</td>
</tr>
<tr>
<td></td>
<td>0.6</td>
<td>$R = -0.0941\varepsilon_z^2 + 0.1006\varepsilon_z + 0.8643$</td>
<td>0.9974</td>
</tr>
<tr>
<td>Al-4Fe$_3$C</td>
<td>0.4</td>
<td>$R = -0.1617\varepsilon_z^2 + 0.1843\varepsilon_z + 0.8535$</td>
<td>0.9908</td>
</tr>
<tr>
<td></td>
<td>0.6</td>
<td>$R = -0.1447\varepsilon_z^2 + 0.1697\varepsilon_z + 0.8539$</td>
<td>0.9930</td>
</tr>
<tr>
<td>Al-4WC</td>
<td>0.4</td>
<td>$R = -0.1294\varepsilon_z^2 + 0.1218\varepsilon_z + 0.8538$</td>
<td>0.9935</td>
</tr>
<tr>
<td></td>
<td>0.6</td>
<td>$R = -0.0859\varepsilon_z^2 + 0.0861\varepsilon_z + 0.8588$</td>
<td>0.9957</td>
</tr>
</tbody>
</table>

Figures 4.4 and 4.5 gives that response of hydrostatic stress against axial strain. The variables in this study are the initial relative density, 0.82 and 0.86, and initial aspect ratio, 0.4 and 0.6. It is important to study hydrostatic stress against axial strain to depict densification behavior of the selected composites. The strength of the powder metallurgy material increases with the increasing density, hence, a higher applied load is required to
further deform the powder metallurgy material each time (Raj et al. 2013). In general all
the graphs follow similar trend. The hydrostatic stress increase at a higher rate as the
material density increases and then it follows a steady state rate. During the initial stage
(0 to 0.4 height strain) a higher hydrostatic stress is required to overcome the yield stress
of the powder metallurgy materials plastic deformation. During this time the larger
number of pores present closes increasing the strength and density of the material thereby
increasing the load requirements. During the later stages (after 0.4 height strain) of
deformation the densification rate is low meaning the effective closure of pores is slow.
The cylindrical pores elongate extensively before it is eliminated are the reason for steady
state stress requirements during later stages when compared to initial stage
(Rajeshkannan et al. 2014).

From Figures 4.4 and 4.5 it is evident that during the initial stage Al-4TiC showed higher
hydrostatic stress, followed by Al-4Fe₃C and Al-4Mo₂C and lowest stress found in Al-
4WC. The same is true during the later stage. Hydrostatic stress mainly due to the friction
between the die and contact surfaces of the specimen plays significant role in pore
closure. Hence, more hydrostatic stress, more densification achieved by the material. The
hydrostatic force depends on porosity, material property and friction condition between
tool and work-piece interface. Lower aspect ratio and higher initial relative density
preform showed better hydrostatic stress when compared to higher aspect ratio preform
and lower initial relative density preform, respectively. Hence, more pore closure in
lower aspect ratio preform and higher initial relative density preforms. This leads to more
densification for these preform as seen in Figures 4.4 and 4.5.
Figure 4.4: Correlation of hydrostatic stress, $\sigma_m / \sigma_{eff}$ and axial strain for initial relative density of 0.86.

Figure 4.5: Correlation of hydrostatic stress, $\sigma_m / \sigma_{eff}$ and axial strain for initial relative density of 0.82.
Similar analysis is presented here on Al-2TiC, Al-2Fe3C, Al-2Mo2C and Al-2WC. A plot has been presented as shown in Fig. 4.6 between true height strain and percentage fractional theoretical density for two different initial aspect ratios, namely, 0.40 and 0.60, these plots being drawn for initial theoretical density of 86%. As seen in Fig. 4.6 the relative density increases up to 0.4 axial strain and thereafter relative density is almost constant till fracture strain except for pure aluminium specimen. Pure aluminium preforms showed better densification rate compared to other aluminium carbide combination, however, the height strain to fracture is very small compared to other composites. Similar to 4% carbide concentration (Figures 4.1-4.2) here also TiC reinforced aluminium showed better densification rate and better final density achieved followed by Fe3C reinforced aluminium and then Mo2C reinforced aluminium. WC reinforced aluminium had the lowest densification rate and final density achieved. Further, Fig. 4.7 gives variation of lateral strain and axial strain for aspect ratio of 0.40 and 0.60, these plots being drawn for starting relative density of 86%. As seen in Fig. 4.7 the lateral deformation is highest in WC reinforced aluminium composite followed by Mo2C, then Fe3C and then TiC composite. Similar behavior found earlier for 4% carbide concentrations. Firstly, the smaller pores present at the center of the preform are eliminated effectively during the deformation process. Secondly, the cylindrical shaped pores and pores towards the side of the preform are extensively elongated and then closed due to the lateral flow of the material during deformation. The second mechanism of pore closure is more in WC reinforced aluminium; hence, it showed lower densification. It is noted that when the carbide concentrations are reduced from 4% (Figures 4.1-4.2) to 2% (Fig. 4.6), the densification performance is improved.
Figure 4.6: Influence of aspect ratios and compositions on the densification behavior during hot deformation for initial relative density of 0.86.

Figure 4.7: The variation of the diameter strain against axial strain during hot deformation for initial relative density of 0.86.
The correlation between relative density, \( R \) and axial strain, \( \varepsilon_z \) for Al-2TiC highlighting the effect of initial aspect ratio of 0.2 and 0.6 and initial relative density of 0.82 and 0.86 is given in Fig. 4.8. From Fig. 4.8 it is noted that there are two stages of deformation. During the first phase of deformation the densification increases rapidly and thereafter the densification remains constant. For a given true height strain, the density attained by a smaller aspect ratio compact is higher when compared to bigger aspect ratio compact. Further, the same is true for higher initial density preforms in comparison to lower initial density preforms. Lower aspect ratio preforms has smaller pore bed height compared to bigger aspect ratio preforms, henceforth, the density attained for any given height strain is higher for lower aspect ratio preform. It is impossible to achieve 100 percent density for powder metallurgy materials and during the first phase of the deformation most of the pores are closed effectively, hence, smaller aspect ratio and bigger initial density compacts has high attained density for any given axial strain. The correlation between relative density, \( R \) and axial strain, \( \varepsilon_z \) of various sintered aluminium composites with aspect ratio of 0.4 and initial relative density of 0.86 is given in Fig. 4.9. From Fig. 4.9 it is noted that for any axial strain, the density attained by the preforms decreases as the number of TiC particles increases in the preforms, however, the strain at fracture is higher for higher content TiC compacts. With increasing hard carbide particles in the preforms, the increasing lateral flow of the material is evident in Fig. 4.10 signaling the increasing strength of the preforms. Hence, more applied load is required for further deformation. Further, with the increasing smaller carbide particles in the preforms increases the number of smaller pores further delaying the densification rate with increasing carbide particles.
Figure 4.8: Correlation of relative density and axial strain highlighting the effect of initial geometry and density of Al-2TiC.

Figure 4.9: Correlation of relative density and axial strain for various sintered aluminium composites.
Figure 4.10: Correlation of $\epsilon_d$ and $\epsilon_z$ for various sintered aluminium composites.

For high strength applications of powder metallurgy parts for industrial applications the strength in the final part needs to be greater than what is available after primary powder metallurgy manufacturing process. It is proved that as the density increases in the powder metallurgy parts the final strength of the part also increases. Apart from reinforcing the powder metallurgy materials with other high strength materials to increase strength one can increase the density to achieve the high strength of powder metallurgy parts. Density in the powder metallurgy parts can be increased by secondary processes such as one carried out in this experiment. One of the major drawbacks during the secondary process is the crack appearing at the visible surfaces. This is due to the pores present in the material some of which is closed in turn increasing the density but some may appear as crack.

Figures 4.11 and 4.12 give the height strain at fracture for respective composites with initial relative density of 0.82 and 0.86. Further, Figures 4.13 and 4.14 highlight the relationship between height strains at fracture against diameter strain at fracture for initial relative density of 0.82 and 0.86. It can be seen that Al-4Mo$_2$C has the highest height strain at fracture followed by Al-4WC, Al-4Fe$_3$C and lowest for Al-4TiC. Further, it can
be seen that higher aspect ratio preforms gives better height strain at fracture compared to lower aspect ratio preforms. Further, from Figures 4.13 and 4.14 it can be depicted that for composites having lower height strain has lower diameter strain as well as in comparison to other composites. As the height stain improves the diameter strain also improves in the aluminium composites tested in this work. These data’s can be properly utilized in selecting the forming parameters and procedures. Further, the data’s can be effectively utilized in die design.

Figure 4.11: Correlation of fracture height strain ($\varepsilon_z$) and composition for initial relative density of 0.86.
Figure 4.12: Correlation of fracture height strain ($\varepsilon_z$) and composition for initial relative density of 0.82.

Figure 4.13: Relationship between fracture height strain ($\varepsilon_z$) and fracture diameter strain ($\varepsilon_d$) for initial relative density of 0.86.
Figure 4.14: Relationship between fracture height strain ($\varepsilon_z$) and fracture diameter strain ($\varepsilon_d$) for initial relative density of 0.82.

Similar plots are presented in Figures 4.15-4.18 for Al-2TiC, Al-2Fe$_3$C, Al-2Mo$_2$C and Al-2WC. As the percentage composition in the composites is increased from 2 percent (Figures 4.15-4.18) to 4 percent (Figures 4.11-4.14) the following observations are seen. Mo$_2$C and Fe$_3$C reinforced aluminium metal matrix composites showed better height strain and diameter strain to fracture whereas TiC and WC reinforced aluminium metal matrix composites showed reduced height strain and diameter strain to fracture. This behavior was found to be same irrespective of aspect ratio and initial relative density. Al-2WC gave the highest height strain to fracture when compared to other compositions. Generally, the effect of varying carbides on the height strain to fracture is more evident in 4 percent carbide concentrations (Figures 4.11 and 4.12) then in 2 percent carbide concentrations (Figures 4.15 and 4.16). Further, higher aspect ratio preforms gave higher height strain to fracture and same is true for all composites. Similar results were found with 4 percent carbide concentrations as seen in Figures 4.11-4.14. Figures 4.17 and 4.18 reveal that height strain at fracture is directly proportional to diameter strain at fracture. The densification rates are found to be lower in Al-4WC, Al-2WC, Al-4Mo$_2$C and Al-2Mo$_2$C when compared to other composites tested here (Figures 4.1 and 4.2) and the
following analysis can be used to select die designs and forming parameters to achieve a good density in these composites at the end of the forging operations.

Figure 4.15: Correlation of fracture height strain ($\varepsilon_z$) and composition for initial relative density of 0.86.
Figure 4.16: Correlation of fracture height strain ($\varepsilon_z$) and composition for initial relative density of 0.82.

Figure 4.17: Relationship between fracture height strain ($\varepsilon_z$) and fracture diameter strain ($\varepsilon_d$) for initial relative density of 0.86.
Figure 4.18: Relationship between fracture height strain ($\varepsilon_z$) and fracture diameter strain ($\varepsilon_d$) for initial relative density of 0.82.

Figures 4.19 and 4.20 shows the microstructural view of the respective composites used in this research work at 0.86 initial relative density (magnification of 100X). Further, Figure 4.19 gives the microstructural views at the center of the specimen and Figure 4.20 gives the microstructural view at the edge of the specimen. The upsetting axis is horizontal in Figures 4.19 and 4.20. Less number of pores is found in the center of the specimen in comparison to the number of pores present at the edge of the specimen. Further, the pores in the center of the specimen are found to be generally smaller in size than the pores at the edge of the specimen. Also the pores appear to be round in shape at the center of the specimen while generally elongated towards the edge of the specimen. These behaviors are found for all the composites. Round and spherical pores do not act as stress risers during application and are assumed to be second phase particles (Rajeshkannan et al. 2014). On the other hand, the elongated pores will appear as cracks when further deformation is employed or may undergo further elongation. This will act as stress risers in actual application and cause failures. Hence, these micrographs can be used effectively to employ repressing activities during forging or help in die design so
that round or spherical pores are left in the final part or the pores are prevented to appear as cracks and are contained within the bulk material.

Figure 4.19: Optical micrographs of various sintered aluminium composites at the center of the specimen.
Figure 4.20: Optical micrographs of various sintered aluminium composites at the edge of the specimen.

4.4 Conclusion

The forming limit and densification behaviors of Al-1TiC, Al-2TiC, Al-3TiC, Al-4TiC, Al-2WC, Al-4WC, Al-2Fe₃C, Al-4Fe₃C, Al-2Mo₂C and Al-4Mo₂C were studied in this research work. The findings are as follows;

- The characteristic nature of the densification curves is similar irrespective of the selected compositions, aspect ratio and initial relative density. Al-4TiC was found to have the highest final density followed by Al-4Fe₃C, Al-4Mo₂C and lowest for Al-4WC. Further, the effect of aspect ratio and initial relative density became less prominent during the final stages of deformation. The Al-2TiC compacts showed better densification thereby the stress formability index of the preform followed
by Al-2Fe$_3$C, then Al-2Mo$_2$C and lowest for Al-2WC compacts. However, Al-2TiC compacts gave lowest axial strain to fracture.

- Al-4Mo$_2$C and Al-4WC have the highest height strain and diameter strain at fracture compared to Al-4Fe$_3$C and Al-4TiC. Also higher aspect ratio preforms showed higher height and diameter strain at fracture. The effect of initial relative density on diameter strain and height strain at fracture was almost negligible for these composites. Decreasing the aspect ratio facilitates uniform deformation resulting in improved densification and formability behavior of the preform, however, limits the height strain to fracture.

- Pores found at the center of the specimen were generally lesser in numbers, smaller in size and round in shape. On the other hand, pores at the edge of the specimens were mainly elongated in the direction of deformation. These behaviors are found for all the composites. The extent of pores and grain structure varies extensively in the composites. The final grain distribution conveys strong alignment along the upsetting direction, causing a fiber structure.

- Pure aluminium preforms major drawback was its very small fracture strain compared to all other composites tested.
Chapter 5
Workability studies of sintered aluminum composites during hot deformation

5.1 Overview
One of the simplest secondary processes used by many researchers (Selvakumar et al. 2007; Taha, El-Mahallawy & El-Sabbagh 2008; Rajeshkannan 2010) is open die forging of cylindrical billets. The residual porosity left in the part causes the failure (visible cracks appearing on the free surface) in the compacts during open die forging and hence, the workability of the materials needs to be studied in the design of forming operation. Workability is a measure of the extent of deformation that a material can withstand due to the induced internal stresses of forming prior to fracture and is not only dependent on the material but also on several forming parameters such as stress and strain rate, friction, temperature, etc (Narayanasamy, Ramesh & Pandey 2005; Rahman & El-Sheikh 1995; El-Domiatry & Shaker 1991). To investigate the workability criteria of the material, ductile fracture criterion for ductile fracture needs be investigated as workability of any material depends mainly on the extent of ductile fracture existent in the material. Over the years, numerous models (Vilotiv et al. 2003, 2006; El-Domiaty 1999) were established to study workability of conventional parts; however, they cannot be directly applied to powder metallurgy parts as the conventional parts are expected to follow volume constancy (Narayanasamy, Ramesh & Pandey 2005; Narayan & Rajeshkannan 2011; Shanmugasundaram & Chandramouli 2009), whereas powder metallurgy parts expected to follow mass constancy (Rahman & El-Sheikh 1995; Hua et al. 2006; Zhang et al. 2000). These essential assumptions were used to develop the plasticity model for respective parts. Many researchers (Spigarelli et al. 2002; Rahimian et al. 2009; Dannininger et al. 1993; Lee et al. 2002; Solhjoo 2009) have studied hot formability of aluminium metal matrix composite where the constitutive equations related to flow stress, temperature, strain rate, flow strain. Kuhn and Downey (1974) proposed a plasticity theory relating yield stress and deformed density by studying the deformation behavior and the plasticity theory of some sintered powder metallurgy materials via open die forging.
Shima & Oyane (1976), Gurson (1977), Vujovic and Shabaik (1986) and Doraivelu et al. (1984) suggested a plasticity theory for porous materials, continuum theory of ductile rupture considering yield criteria for porous material and a novel yield criterion for porous material certifying it with experiments and simulations, respectively. A plasticity theory proposed by Shima and Oyane (1976) and Green (1972) has been utilized to study workability and deformation behavior considering numerous spherical cracks and voids, stress in the direction of compression and relative density. Narayanasamy, Senthilkumar & Pandey (2007), Narayanasamy, Ponalagusamy & Subramanian (2001) and Narayanasamy, Anandakrishnan & Pandey (2008) studied the fracture criterion of porous materials under plane stress state, uniaxial stress state and triaxial stress state conditions relating hoop stress, mean stress, effective stress and the several stress ratio parameters. Narayanasamy, Ramesh & Pandey (2006) investigated the formability characteristics of Al-Al2O3 MMC’s using several stress state situations, namely uniaxial, plane and triaxial stress conditions. The effects of different curve fitting techniques, preform geometry, and initial relative density on the workability criteria were presented. They used a general yield criterion considering an-isotropic parameters for powder metallurgy metals which were earlier proposed by Narayanasamy, Ponalagusamy & Subramanian (2001) presented a novel flow rule with an-isotropic factors for porous metals. One of the most important parameters in the study of workability characteristics is the formability stress index proposed by Rahman and El-Sheikh (1995). It describes the effect of hydrostatic stress and the effective stress on the powder metallurgy compacts. Ko, Park & Yoo (1999) studied the microstructure and hot workability of SiCp/AA 2024 composite and reported that dynamic recrystallization was responsible for the hot restoration of the composites. They showed the addition of the SiCp to an AA2024 matrix alloy increased the dislocation density, resulting in a high flow stress and a low critical strain for the dynamic recrystallization of the composite. Further, they reported upon increasing the SiCp volume fraction, the flow stress increased and the failure strain decreased.

Narayanasamy, Senthilkumar & Pandey (2008) studied hot forging of powder metallurgy sintered high strength 4% titanium carbide composite steel preforms under different stress state conditions proposing a new geometrical shape factor and exponential
relationship between the relative density ratio and hoop strain. It was reported that a straight line relationship was established between relative density against new geometrical shape factor and respective stress ratio parameters. Rajeshkannan (2010) studied workability of sintered copper alloy preforms during cold upsetting using different curve fitting techniques and reported that decreasing aspect ratio facilitated deformation and improved the formability stress index, however, limited height strain to fracture. Similar study was carried out by Narayanasamy, Senthilkumar & Pandey (2006) via hot forging of 4% titanium carbide composite steels preforms and similar results were reported. Taha, El-Mahallawy and El-Sabbagh (2008) presented some experimental data on workability of aluminium-particulate-reinforced metal matrix composites prepared by stir-casting, squeeze-casting and powder metallurgy techniques. They reported that the workability of aluminium SiC and Al2O3 reinforced metal matrix composites is affected positively by the following: applying intermediate heat treatment, decreasing particulate volume fraction, decreasing particulate size and reinforcing with SiC in a wrought alloy matrix rather than Al2O3.

Gouveia, Rodrigues & Martins (2000) conducted experiments on powder metallurgy compacts of several geometries and reported that the initiation of the ductile fracture can be predicted. The behavior of hoop stress, hydrostatic stress and axial stress of any powder metallurgy materials is vital in all forging processes and Narayanamurti, Nageswara & Kashyap (2004) offered some of the significant standards normally used for the calculation of ductile fracture. Rajeshkannan and Narayan (2013) showed smaller height to diameter ratio compacts showed constant densification because of quick stress transfer between powder particles causing excessive matrix and geometric hardening. Rao & Hawbolt (1992) obtained flow stress in terms of process variables, strain, strain rate and temperature using computer controlled thermo-mechanical simulator. Bao (2005) published a correlation between two important parameters, the stress triaxiality and equivalent strain to study the failure of powder metallurgy materials, that is, development of crack during deformation. Sowerby et al. (1984) reported on the effect of the hoop strain and axial strain at the free surface of the powder metallurgy compacts and respective stresses were determined. Kumar, Narayanasamy and Loganathan (2012)
investigated the ductile fracture in metal working experimentally and validated it using theoretical models. They showed some of the significant criterions normally employed for the prediction of ductile rupture under several stress state conditions, namely, plane, uniaxial and triaxial stress state.

The residual porosity left in the powder metallurgy parts after the primary powder metallurgy process is a major drawback and these residual porosities are the main cause of failure during the secondary process and hence, workability studies are important in the preform geometry design and die constraint designs. Deformation control to avoid fracture can be established by careful selection of process parameters, the factors being die shaping, lubrication, preform shape, preform dimensions and density. Geometrical design of the preform in metal forging of complex parts has great effects on the forging load and plays a key role in improving product quality, such as ensuring defect-free product and proper metal flow. By careful selection of process parameter and deformation process densification can be enhanced and crack appearance on the deforming preforms can be avoided by increasing the compressive level of stresses on the material (Lee et al. 2002; Sedighi & Tokmechi 2008; Butuc, Gracio & Rocha 2006).

Recently, hot deformation has been considered by numerous researchers due to the growing request of powder metallurgy hot forged parts such as connecting rods in automobiles, camshafts for automobile engines and aircraft and industrial cutting and forming tools (Kandavel, Chandramouli & Shanmugasundaram 2009; Kim 2002; Szczepanik & Sleboda 1996; Eghbali & Abdollah-Zadeh 2006; Farnoush et al. 2010). The material movement throughout hot forging is complex as the hardening and softening mechanisms are affected by the temperature and strain rate (Solhjoo 2009). Since the chief reason of fracture in forging is the circumferential tensile stresses, it is therefore vital to explore fracture during hot forging of sintered powder materials (Narayanasamy & Selvakumar 2005; Rajeshkanna et al. 2008). The forming limit of powder materials is a matter of great concern and the workability of metals is an essential factor in designing of forming operations (Zhang et al. 2009; Ramesh & Senthivelan 2010). In the present investigation the hot workability studies is carried out to understand the forming behavior
of the selected compositions, Al-1TiC, Al-2TiC, Al-3TiC, Al-4TiC, Al-1Fe3C, Al-2Fe3C, Al-4Fe3C, Al-6Fe3C, Al-1Mo2C, Al-2Mo2C, Al-3Mo2C, Al-4Mo2C, Al-1WC, Al-2WC, Al-3WC and Al-4WC.

5.2 Results and discussions

A plot has been presented as shown in Fig. 5.1 between stress ratio, $\sigma_\theta / \sigma_{eff}$, and relative density, R, for two different initial aspect ratios, namely, 0.40 and 0.60, these plots being drawn for initial relative density of 86%. Further, similar plots have been plotted for stress ratios $\sigma_n / \sigma_{eff}$ and $\sigma_z / \sigma_{eff}$ presented in Figures 5.2 and 5.3. From these plots it can be seen the effect of aspect ratio and composition is nil on the characteristics behavior. The respective stress ratio parameters increase as the relative density increase. The stress ratio behavior can be found in two stages against densification, one from the start of densification till 0.912 relative density and the other from 0.912 relative density till the end of deformation. The slope is found to increase from stage one to stage two as high load is required to further deform the specimen as very little pores are left towards the end of deformation. Against densification, the highest stress ratio value was achieved by TiC content composite followed by Fe3C composite, Mo2C composite and lowest was WC containing composite. Further, the highest stress ratio parameter was obtained for smaller aspect ratio preform, which is true for all stress ratio preforms. This indicates that WC containing composite and higher aspect ratio preform has further chance to be deformed provided preform is free of cracks. The lateral deformation at the contact surface is lowest in TiC composite as seen in Fig. 4.3 for any given height strain indicating the bulging phenomenon is highest in TiC composite compared to Fe3C, Mo2C, and WC composites. Same can be seen in Table 5.1. Hence, the hoop stress is found to be highest in TiC composite compared to other composites. The same is true for the hydrostatic stress ($\sigma_m / \sigma_{eff}$) found to be highest in TiC composite.
Figure 5.1: Relationship between stress ratio \( \left( \frac{\sigma_\theta}{\sigma_{\text{eff}}} \right) \) and relative density during hot deformation for initial relative density of 0.86.

Figure 5.2: Relationship between stress ratio \( \left( \frac{\sigma_m}{\sigma_{\text{eff}}} \right) \) and relative density during hot deformation for initial relative density of 0.86.
Figure 5.3: Relationship between stress ratio ($\sigma_z / \sigma_{eff}$) and relative density during hot deformation for initial relative density of 0.86.

Table 5.1: Actual bulged length for the respective composites at 0.40 height strain

<table>
<thead>
<tr>
<th>Composite</th>
<th>Bulge length (x)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-4TiC</td>
<td>0.4 1.052 mm</td>
</tr>
<tr>
<td></td>
<td>0.6 1.460 mm</td>
</tr>
<tr>
<td>Al-4Fe$_3$C</td>
<td>0.4 1.037 mm</td>
</tr>
<tr>
<td></td>
<td>0.6 1.394 mm</td>
</tr>
<tr>
<td>Al-4Mo$_2$C</td>
<td>0.4 0.980 mm</td>
</tr>
<tr>
<td></td>
<td>0.6 1.355 mm</td>
</tr>
<tr>
<td>Al-4WC</td>
<td>0.4 1.020 mm</td>
</tr>
<tr>
<td></td>
<td>0.6 1.305 mm</td>
</tr>
</tbody>
</table>

Similar analysis is presented here on Al-2TiC, Al-2Fe$_3$C, Al-2Mo$_2$C and Al-2WC. A plot has been presented as shown in Fig. 5.4 between stress ratio, $\sigma_\theta / \sigma_{eff}$, and relative density, R, for two different initial aspect ratios, namely, 0.40 and 0.60, these plots being
drawn for initial theoretical density of 86%. Further, similar plots have been plotted for stress ratios $\sigma_m/\sigma_{eff}$ and $\sigma_z/\sigma_{eff}$ presented in Figures 5.5 and 5.6. From these plots it can be seen that the influence of aspect ratio and composition is nil on the characteristics behavior. The hoop, mean and axial stresses increase as the relative density increase. The rate, at which the hoop, mean and axial stresses increase, increases as the densification progresses and the final stress ratio and relative density achieved at the end of deformation is different for the respective composition. Against densification, the highest stress value was achieved by TiC content composite followed by Fe₃C composite, Mo₂C composite and lowest was WC containing composite. Further, the highest stress was obtained for smaller aspect ratio preform, which is true for all stress ratio preforms. As the carbide concentrations are decreased from 4% (Figures 5.1-5.3) to 2% (Figures 5.4-5.6), the characteristics behavior, trend and the effect of aspect ratio is unchanged. However, carbide concentrations of 2% showed higher stress ratio parameters compared to carbide concentrations of 4%.

Figure 5.4: Relationship between hoop stress ($\sigma_θ/\sigma_{eff}$) and relative density during hot deformation for initial relative density of 0.86.
Figure 5.5: Relationship between mean stress ($\sigma_m / \sigma_{eff}$) and relative density during hot deformation for initial relative density of 0.86.

Figure 5.6: Relationship between axial stress ($\sigma_z / \sigma_{eff}$) and relative density during hot deformation for initial relative density of 0.86.
Table 5.2: Actual bulged length for the respective composites at 0.40 height strain

<table>
<thead>
<tr>
<th>Composite</th>
<th>Bulge length (x)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-2TiC</td>
<td>0.4 1.060 mm</td>
</tr>
<tr>
<td></td>
<td>0.6 1.494 mm</td>
</tr>
<tr>
<td>Al-2Fe₃C</td>
<td>0.4 0.942 mm</td>
</tr>
<tr>
<td></td>
<td>0.6 1.480 mm</td>
</tr>
<tr>
<td>Al-2Mo₂C</td>
<td>0.4 1.055 mm</td>
</tr>
<tr>
<td></td>
<td>0.6 1.378 mm</td>
</tr>
<tr>
<td>Al-2WC</td>
<td>0.4 1.044 mm</td>
</tr>
<tr>
<td></td>
<td>0.6 1.320 mm</td>
</tr>
</tbody>
</table>

Figures 5.7 to 5.9 is drawn for hoop \(\sigma_\theta / \sigma_{\text{eff}}\), mean \(\sigma_m / \sigma_{\text{eff}}\), and axial \(\sigma_z / \sigma_{\text{eff}}\) stress to that of relative density respectively for various sintered aluminium composite with aspect ratio of 0.40 and initial theoretical density of 86% under hot upsetting. The behavior of the composite plotted in Figures 5.7 to 5.9 was found to be identical. It can be seen that the effect of TiC content in the aluminium composite made nil effect on the stress ratio behavior. However, the maximum stress ratio is achieved by the lowest percentage of TiC containing composite, which is true for all the stress ratio parameters. Hence, Al-4TiC composite can be deformed more if it is free of defects. Increasing the TiC particles in the preforms, increases chances of further deformation, promoting densification and strength in the composite. It is also noted that the rate at which the stress ratio parameters increase, increases as the deformation progresses.
Figure 5.7: Correlation of $\sigma_\theta / \sigma_{\text{eff}}$ and relative density for various sintered aluminium composites.

Figure 5.8: Correlation of $\sigma_m / \sigma_{\text{eff}}$ and relative density for various sintered aluminium composites.
Figure 5.9: Correlation of $\sigma_z/\sigma_{eff}$ and relative density for various sintered aluminium composites.

A plot has been presented as shown in Fig. 5.10 between stress ratio, $\sigma_\theta/\sigma_{eff}$, and axial strain for two different initial aspect ratios, namely, 0.40 and 0.60, these plots being drawn for initial relative density of 86%. Further, similar plots have been plotted for stress ratios $\sigma_m/\sigma_{eff}$ and $\sigma_z/\sigma_{eff}$ presented in Figures 5.11 and 5.12. The effect of aspect ratio on the stress ratios, $\sigma_\theta/\sigma_{eff}$, $\sigma_m/\sigma_{eff}$, and $\sigma_z/\sigma_{eff}$ is literally nil when plotted against axial strain except for TiC containing composite. The stress ratio parameters rises to a maximum value as the deformation starts and then settles for the steady state stress for the rest for the deformation. However, all the stress ratios slightly lower towards the end mainly in Al-4Mo$_2$C and Al4-WC. For any given height strain, the hoop, mean and axial stress obtained is highest for TiC containing compacts, followed by Fe$_3$C, Mo$_2$C and lowest for WC containing compacts. The TiC particulates impede the motion of dislocations more than Fe$_3$C, Mo$_2$C and WC particulates and hence the stress required for further plastic deformation for TiC composite is more than other composite. Due to this reason the stress ratios, $\sigma_\theta/\sigma_{eff}$, $\sigma_m/\sigma_{eff}$, and $\sigma_z/\sigma_{eff}$, are
higher for TiC composite for any given true height strain. Further, the polynomial curve fitting results with correlation values close to 1.0 for $\sigma_{\theta}/\sigma_{\text{eff}}$ vs $\varepsilon_z$ are given in Table 5.3. It is found that the first order values decrease whilst the second order values increase with the increasing aspect ratio. Also the first order value which increases the hoop stress linearly are found to be higher in TiC composite compared to other composites.

Figure 5.10: Relationship between stress ratio ($\sigma_{\theta}/\sigma_{\text{eff}}$) and axial strain during hot deformation for initial relative density of 0.86.
Figure 5.11: Relationship between stress ratio ($\sigma_{\text{eff}}/\sigma_{\text{m}}$) and axial strain during hot deformation for initial relative density of 0.86.

Figure 5.12: Relationship between stress ratio ($\sigma_{\text{eff}}/\sigma_{\text{eff}}$) and axial strain during hot deformation for initial relative density of 0.86.
Table 5.3: Polynomial curve fitting result – $\sigma_0/\sigma_{\text{eff}}$ vs $\varepsilon_z$ for initial relative density of 0.86.

<table>
<thead>
<tr>
<th>Material</th>
<th>Aspect Ratio</th>
<th>Equation</th>
<th>$R_c^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-4TiC</td>
<td>0.4</td>
<td>$\sigma_0/\sigma_{\text{eff}} = -2.3226\varepsilon_z^2 + 2.3946\varepsilon_z + 0.7555$</td>
<td>0.9989</td>
</tr>
<tr>
<td></td>
<td>0.6</td>
<td>$\sigma_0/\sigma_{\text{eff}} = -2.209\varepsilon_z^2 + 2.3554\varepsilon_z + 0.5968$</td>
<td>0.9922</td>
</tr>
<tr>
<td>Al-4Mo$_2$C</td>
<td>0.4</td>
<td>$\sigma_0/\sigma_{\text{eff}} = -1.183\varepsilon_z^2 + 1.0494\varepsilon_z + 0.7852$</td>
<td>0.9991</td>
</tr>
<tr>
<td></td>
<td>0.6</td>
<td>$\sigma_0/\sigma_{\text{eff}} = -0.5314\varepsilon_z^2 + 0.5667\varepsilon_z + 0.8191$</td>
<td>0.9951</td>
</tr>
<tr>
<td>Al-4Fe$_3$C</td>
<td>0.4</td>
<td>$\sigma_0/\sigma_{\text{eff}} = -1.138\varepsilon_z^2 + 1.3437\varepsilon_z + 0.6908$</td>
<td>0.9949</td>
</tr>
<tr>
<td></td>
<td>0.6</td>
<td>$\sigma_0/\sigma_{\text{eff}} = -1.0076\varepsilon_z^2 + 1.1931\varepsilon_z + 0.7070$</td>
<td>0.9920</td>
</tr>
<tr>
<td>Al-4WC</td>
<td>0.4</td>
<td>$\sigma_0/\sigma_{\text{eff}} = -0.9698\varepsilon_z^2 + 0.9169\varepsilon_z + 0.7061$</td>
<td>0.9950</td>
</tr>
<tr>
<td></td>
<td>0.6</td>
<td>$\sigma_0/\sigma_{\text{eff}} = -0.7433\varepsilon_z^2 + 0.7681\varepsilon_z + 0.7089$</td>
<td>0.9961</td>
</tr>
</tbody>
</table>

Similar analysis is presented here on Al-2TiC, Al-2Fe$_3$C, Al-2Mo$_2$C and Al-2WC. A plot has been presented as shown in Fig. 5.13 between stress ratio, $\sigma_0/\sigma_{\text{eff}}$, and true height strain for two different initial aspect ratios, namely, 0.40 and 0.60, these plots being drawn for initial theoretical density of 86%. Further, similar plots have been plotted for stress ratios $\sigma_m/\sigma_{\text{eff}}$ and $\sigma_z/\sigma_{\text{eff}}$ presented in Figures 5.14 and 5.15. It can be seen from Fig. 5.13 that the hoop stress generated in the pure aluminium preform is very similar to the Al-2TiC composite for any given height strain. The influence of aspect ratio on hoop, mean and axial stresses is literally nil, especially towards the end of deformation when plotted against axial strain. The hoop, mean and axial stresses rises to a maximum value as the deformation starts and then settles for the steady state stress for the rest for the deformation. For any given axial strain, the hoop, mean and axial stresses obtained are highest for TiC containing compacts, followed by Fe$_3$C, Mo$_2$C and lowest for WC containing compacts. Similarly here the reduction in carbide concentrations from 4% to 2% has no effect on the characteristics behavior, trend and on the effect of aspect ratio. However, lower carbide concentration preforms produced higher stress ratios for any given height strain.
Figure 5.13: Relationship between hoop stress ($\sigma_\theta / \sigma_{eff}^\theta$) and axial strain during hot deformation for initial relative density of 0.86.

Figure 5.14: Relationship between mean stress ($\sigma_m / \sigma_{eff}^m$) and axial strain during hot deformation for initial relative density of 0.86.
Figure 5.15: Relationship between axial stress \( (\sigma_z / \sigma_{\text{eff}}) \) and axial strain during hot deformation for initial relative density of 0.86.

The correlation between hoop stress, \( \sigma_\theta / \sigma_{\text{eff}} \) and axial strain, \( \varepsilon_z \) for Al-2TiC highlighting the effect of initial aspect ratio of 0.2 and 0.6 and initial relative density of 0.82 and 0.86 is given in Fig. 5.16. Further, the correlation between \( \sigma_\theta / \sigma_{\text{eff}} \) and \( \varepsilon_z \) of various sintered aluminium composites with aspect ratio of 0.4 and initial relative density of 0.86 is given in Fig. 5.17. It is noted from Figures 5.16 & 5.17 that the hoop stress on the cylindrical preforms during hot deformation increases sharply to almost a maximum value with the increase in strain followed by attainment of steady state stress. The curves plotted in Figures 5.16 and 5.17 clearly portrays the influence of initial density, geometry and percentage of TiC in the preforms on the maximum stress achieved for any given axial strain and the final steady state stress. As expected, the parameter, \( \sigma_\theta / \sigma_{\text{eff}} \), increases with increasing levels of strain and increasing initial density and decreasing aspect ratio. It is found that the parameter, \( \sigma_\theta / \sigma_{\text{eff}} \), decreases for any given axial strain as the TiC particles in the aluminium composite is increased. To achieve same hoop stress in the aluminium composites with varying TiC contents, the higher TiC containing
composite needs to be deformed more than the lower TiC containing composite. This suggests that the aluminium composite strength improves with the increasing TiC particles in the composites. The same is true for stress ratio parameters $\sigma_{\theta}/\sigma_{\text{eff}}$ and $\sigma_z/\sigma_{\text{eff}}$ as seen in Figures 5.18 and 5.19 as the variation amongst axial, hoop and hydrostatic stress is almost negligible against axial strain.

![Figure 5.16](image)

Figure 5.16: Correlation of $\sigma_{\theta}/\sigma_{\text{eff}}$ and $\epsilon_z$ of Al-2TiC highlighting the effect of initial geometry and density.
Figure 5.17: Correlation of $\sigma_\theta / \sigma_{\text{eff}}$ and $\varepsilon_z$ for various sintered aluminium composites.

Figure 5.18: Correlation of $\sigma_w / \sigma_{\text{eff}}$ and $\varepsilon_z$ for various sintered aluminium composites.
A plot has been presented as shown in Fig. 5.20 between formability index, $\beta$, and axial strain for two different initial aspect ratios, namely, 0.40 and 0.60, these plots being drawn for initial relative density of 86%. The formability of the TiC containing compacts was found to be higher followed by Fe$_3$C and Mo$_2$C composites. WC containing compacts showed lowest formability ratio for any given axial strain. For any given axial strain, the relative density is found to be highest in TiC composite, Fig. 4.1, meaning the porosity level is low and hence the reason for high formability index for TiC composite. The true hydrostatic stress ($\sigma_m / \sigma_{\text{eff}}$) is higher in TiC composite than the true effective stress compared to Fe$_3$C, Mo$_2$C and WC composite (Fig. 4.4) is the reason for high formability in TiC composite. The compositions are calculated using weight percentage and TiC particulate being the lowest weight followed by Fe$_3$C, Mo$_2$C and WC (Table 4.1). This means the amount of smaller and fine pores present in Al-4TiC composite is more than other composite. The effective closure of pores is more in TiC composite.
during hot upsetting increasing the densification (Fig. 4.1) and hence, for the same reason the formability ratio is higher in TiC composite.

The polynomial curve fitting results with correlation values close to 1.0 for $\beta$ vs $\varepsilon_x$ are given in Table 5.4. Second order and third order polynomial fits are used to obtained the correlation values near unity, however, the constant, first and second order values are up most important. A constant formability stress value was obtained for zero axial strain. This constant value is approximated to be $2.0 \pm 0.3$. It is found that the first order values increase while the second order values and constant values decrease with the increasing aspect ratio and hence the formability of the material decreases with increasing aspect ratio. The first order values are found to be positive whilst the second order values are found to be negative revealing that at the initial stages of deformation for low enhancement in axial strain the formability index is profound.

Further, a plot has been presented as shown in Fig. 5.21 between formability index, $\beta$, and relative density for two different initial aspect ratios, namely, 0.40 and 0.60, these plots being drawn for initial relative density of 86%. The effect of aspect ratio and composition showed nil effect on the formability behavior against relative density, however, the final formability ratio achieved against relative density and axial strain is also important. In view of this a graph of formability index at fracture and axial strain at fracture is plotted as shown in Fig. 5.22. The axial strain at fracture is found to be higher for higher aspect ratio preforms in comparison to lower aspect ratio preforms. There is an inverse relationship between fracture strain and fracture formability stress index as shown in Fig. 5.22. This effect is more in lower aspect ratio preforms. WC containing compacts showed higher fracture strain followed by Mo$_2$C composite, then Fe$_3$C composite and lowest fracture strain for TiC composites. Also the number of carbide particles is higher in Al-4TiC composite compared to other composites prepared meaning the amount of pores is higher in Al-4TiC leading to lower fracture strain.
Figure 5.20: Relationship between formability stress ratio and axial strain during hot deformation for initial relative density of 0.86.

Table 5.4: Polynomial curve fitting result – $\beta$ vs $\varepsilon_z$ for initial relative density of 0.86.

<table>
<thead>
<tr>
<th>Al-4TiC</th>
<th>0.4</th>
<th>$\beta = -7.1569\varepsilon_z^2 + 7.2373\varepsilon_z + 2.2798$</th>
<th>$R_c^2 = 0.9979$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.6</td>
<td>$\beta = -7.5964\varepsilon_z^2 + 7.4891\varepsilon_z + 1.7654$</td>
<td>$R_c^2 = 0.9971$</td>
</tr>
<tr>
<td>Al-4Mo$_2$C</td>
<td>0.4</td>
<td>$\beta = -3.4469\varepsilon_z^2 + 2.9723\varepsilon_z + 2.3094$</td>
<td>$R_c^2 = 0.9965$</td>
</tr>
<tr>
<td></td>
<td>0.6</td>
<td>$\beta = +3.8739\varepsilon_z^3 - 7.7228\varepsilon_z^2 + 4.3776\varepsilon_z + 2.1973$</td>
<td>$R_c^2 = 0.9943$</td>
</tr>
<tr>
<td>Al-4Fe$_3$C</td>
<td>0.4</td>
<td>$\beta = -3.3318\varepsilon_z^2 + 3.8454\varepsilon_z + 2.1501$</td>
<td>$R_c^2 = 0.9928$</td>
</tr>
<tr>
<td></td>
<td>0.6</td>
<td>$\beta = +4.6303\varepsilon_z^3 - 9.5042\varepsilon_z^2 + 6.0699\varepsilon_z + 1.9044$</td>
<td>$R_c^2 = 0.9913$</td>
</tr>
<tr>
<td>Al-4WC</td>
<td>0.4</td>
<td>$\beta = +3.1557\varepsilon_z^3 - 7.1485\varepsilon_z^2 + 4.5426\varepsilon_z + 1.8638$</td>
<td>$R_c^2 = 0.9931$</td>
</tr>
<tr>
<td></td>
<td>0.6</td>
<td>$\beta = +9.71\varepsilon_z^3 - 15.516\varepsilon_z^2 + 7.4421\varepsilon_z + 1.6901$</td>
<td>$R_c^2 = 0.9963$</td>
</tr>
</tbody>
</table>
Figure 5.21: Relationship between formability stress ratio and relative density during hot deformation for initial relative density of 0.86.

Figure 5.22: Relationship between fracture strain ($\varepsilon_f$) and formability stress index at fracture ($\beta_f$) during hot deformation for initial relative density of 0.86.
Similar analysis is presented here on Al-2TiC, Al-2Fe₃C, Al-2Mo₂C and Al-2WC. A plot has been presented as shown in Fig. 5.23 between formability stress ratio, $\beta$, and true height strain for two different initial aspect ratios, namely, 0.40 and 0.60, these plots being drawn for initial theoretical density of 86%. The formability index for pure aluminium is higher than any other aluminium carbide composites tested; however, it has very low height strain to fracture. The behavior of pure aluminium is very close to Al-2TiC composite. The formability of the TiC containing compacts was found to be higher followed by Fe₃C and Mo₂C composites. WC containing compacts showed lowest formability ratio for any given axial strain. For any given axial strain, the relative density is found to be highest in TiC composite, Fig. 4.2, meaning the porosity level is low and hence the reason for high formability ratio for TiC composite. Further, the true mean and hoop stress is higher in TiC composite than the true effective stress compared to Fe₃C, Mo₂C and WC composite (Figures 5.14 and 5.13) is the reason for high formability in TiC composite. Further, a plot has been presented as shown in Fig. 5.24 between formability stress ratio, $\beta$, and relative density for two different initial aspect ratios, namely, 0.40 and 0.60, these plots being drawn for initial theoretical density of 86%. Similarly, the effect of aspect ratio and composition showed nil effect on the formability behavior against relative density, however, the final formability ratio achieved against density and axial strain is also important. Further, in Fig. 5.25 similar behavior is seen as in Fig. 5.22. WC containing compacts showed higher fracture strain followed by Mo₂C composite, then Fe₃C composite, then TiC composite and lowest fracture strain for pure aluminium preform. Generally, it is seen that when the carbide concentrations are reduced from 4% (Fig. 5.22) to 2% (Fig. 5.25), the formability stress index at fracture is increased. However, it reduced the axial strain at fracture for most cases. Theses graphs are important and can be utilized in the preform shape and die constraint design.
Figure 5.23: Relationship between formability stress ratio and axial strain during hot deformation for initial relative density of 0.86.

Figure 5.24: Relationship between formability stress ratio and relative density during hot deformation for initial relative density of 0.86.
Figure 5.25: Relationship between fracture strain ($\varepsilon^f_z$) and formability stress index at fracture ($\beta^f$) during hot deformation for initial relative density of 0.86.

The correlation between formability stress index, $\beta$ and axial strain, $\varepsilon_z$ for Al-2TiC highlighting the effect of initial aspect ratio of 0.2 and 0.6 and initial relative density of 0.82 and 0.86 is given in Fig. 5.26. Further, the correlation between $\beta$ and $\varepsilon_z$ of various sintered aluminium composites with aspect ratio of 0.4 and initial relative density of 0.86 is given in Fig. 5.27. It is noted that formability stress index varies with initial theoretical density, aspect ratio and percentage of TiC content in the preforms. The formability stress index increases with the increasing axial strain. Further, as the initial theoretical density increases and aspect ratio decreases, the formability stress index also increases for any given axial strain. Also as the percentage of TiC increases in the preforms, the formability stress index decreases for any given axial strain. Formability stress index determines the fracture limit of the deforming material and it can be seen with the increasing percentage of TiC in the preforms, increasing pore bed height and decreasing initial theoretical density decreases the workability of the materials. With increasing smaller TiC hard carbide particles taking up space between Al particles in the preform
increases the number of smaller pores giving rise to more lateral deformation (Fig. 4.7) which in turn reduces the formability of the material.

Figure 5.26: Correlation of formability stress index, $\beta$ and axial strain of Al-2TiC highlighting the effect of initial geometry and density.
Figure 5.27: Correlation of formability stress index, $\beta$ and axial strain for various sintered aluminium composites.

Experimental data obtained during the hot deformation of TiC containing aluminium composite has been utilized to plot fracture strain ($\varepsilon_z'\varepsilon$) and formability stress index at fracture ($\beta'$) as shown in Fig. 5.28. It is noted that the fracture axial strain increases with the increasing percentage of TiC content in the composites; however, the formability of the material is reduced. This plot can be effectively used for the design of part geometry and die constraints at the free end of the deforming compact.
Figure 5.28: Correlation of $\varepsilon_z$ at fracture and formability stress index at fracture for various sintered aluminium composites.

Further, to understand the deformation behavior of Al-4TiC, Al-4Fe$_3$C, Al-4Mo$_2$C, and Al-4WC, the microstructure view of 100X magnification is shown in Figures 5.29 and 5.30, respectively. Particularly, the view was selected one at the center (Fig. 5.29) and other one at the extreme diametric side of each of the preforms (Fig. 5.30) in order to view the presence of porosities. The upsetting axis is horizontal in Figures 5.29 and 5.30. The grain boundaries are clear and straight. As seen in Fig. 5.29, less number of pores is found in TiC and Fe$_3$C when compared to Mo$_2$C and WC composites, however, more equi-axed grains are found in Mo$_2$C and WC composites compared to TiC and Fe$_3$C composites. It can be seen the reduction in pores from undeformed specimen (green compact) to medium deformed and full deformed (at fracture) in Fig. 5.30. The grains are equi-axed form in the green compact, Fig. 5.30 (a), and then during deformation process the grains elongate in the direction perpendicular to the compression direction as seen in Fig. 5.30 (c). The pores at the center of the specimen are found to be spherical shape (Fig. 5.29) whilst at the diametric ends are mainly elongated (Fig. 5.30).
Figure 5.29: Optical micrographs of various sintered aluminium composites with 50% deformation at the center.
Figure 5.30: Optical micrographs of Al-4TiC at the diametrical end: a) undeformed, b) medium deformed and c) fully deformed.

Similar analysis is presented here on Al-2TiC, Al-2Fe₃C, Al-2Mo₂C and Al-2WC. Figure 5.31 show the optical micrographs of hot forged aluminium composites along the upsetting direction. It can be seen that the grains are deformed and elongated perpendicular to the compression direction (compression direction is horizontal in Fig. 5.31). The elongation of particles is more in WC, followed by Mo₂C, Fe₃C and lowest in TiC composite. The inter-particle spacing in TiC reinforced aluminium composite is small due to more TiC particles present then in Fe₃C, Mo₂C, and WC reinforced aluminium, respectively. Hence, the effective closure of pores is more in Al-2TiC
composite promoting densification and formability as seen in Figures 4.6 and 5.23, respectively.

Al-2TiC                         Al-2Fe₃C
Al-2Mo₂C                        Al-2WC

Figure 5.31: Optical micrographs of various sintered aluminium composites with 50% deformation at the center.

Figure 5.32 presents the optical micrographs of aluminium MMC’s containing 1%, 2%, 3% and 4% TiC particles along the upsetting direction. Upon increasing the TiC content in the composite, the inter-particle space amongst the powders reduces and the matrix grains change from elongated to equi-axed form. With the increasing TiC particles in the aluminium composite preforms, the particles have axis of approximately same length, thus more planes on which to slip promoting strength in the preform. Figure 5.33 show the microstructure of material consisting of 3% TiC at 100X magnification. The number of pores found at the center of the specimen is lower when compared to the number of
pores present at the edge of the specimen. Further, at the center of the specimen the pores are generally round in shape while at the edge of the specimen it is generally elongated along the direction of material flow. Hence, it is noted for cylindrical compacts the pores get eliminated fully more at the center of the specimen than at the edge of the specimen during the secondary deformation process.

Figure 5.32: Optical micrographs of respective MMC’s.
Figure 5.33 (a) and (b): Optical micrographs at centre and diametric extreme of the Al-3TiC preform, respectively.

5.3 Conclusion

The design of preform shape and die are very important such that the final part produced is free from defects (fracture) since there is little possibility that the cracks can be arrested during the repressing stage of the deformation. Accordingly the major conclusions have been drawn that are as follows.

- The variation of aspect ratio and hard carbide particles in aluminium composite made nil impact on the trend of stress ratio behavior against densification, however, against true axial strain induced is prominent.

- TiC reinforced aluminium metal matrix composites produced the highest mean, hoop and hydrostatic stresses, followed by Fe$_3$C, then Mo$_2$C and lowest for WC reinforced aluminium metal matrix composites.

- The relationship between stress ratio and formability against axial strain and relative density is established using polynomial curve fitting results. The formability of TiC reinforced composite was better than other composites while WC reinforced composite showed very poor formability.
• The formability of the composites was found to improve with increasing initial relative density and reducing aspect ratio. Increasing the TiC content from 1% to 4% reduced the density thereby reducing stress formability index.

• The amount of pores and grain structure varies significantly in the composites tested at 50% deformation. The final grain distribution reveals strong orientation along the compression direction, resulting in a fiber structure. The higher TiC content preform had lower inter-particle spacing and had equi-axed matrix grains compared to elongated matrix grains for lower TiC content preforms.
Chapter 6
Workability limit diagram

6.1 Overview
Numerous researches establish using Oyane’s fracture principle to frame the mathematical model and matched with experimental outcomes showing good agreement (Vilotiv et al. 2006; Shabara, El-Domiaty & Kandil 1996; Zhang et al. 2009). Oyane’s fracture principle states formability stress factor and the strain that has been effectively combined to create the workability limits for wrought parts (Vilotiv et al. 2003, 2006). It is presented (Vilotiv et al. 2003, 2006; El-Domiaty 1999) that the idea of workability limit can be useful to account for the effect of formability stress factor and define the strain to fracture at any material point where the formability stress factor is constant throughout the process. For powder metallurgy parts this condition is not fulfilled (Narayanasamy, Ramesh & Pandey 2005; Rahman & El-Sheikh 1995; Narayanasamy, Anandakrishnan & Pandey 2008) and as projected in (Vilotiv et al. 2003, 2006), a workability limit should be supplemented with a fracture criterion to take into account the path-dependence of fracture. The fracture criterion used in the workability limit (characterized by effective strain against formability stress factor at fracture) can be expressed by integrating formability stress factor over the strain path instead of experimental values. The current study is intended to develop a mathematical model using Oyane’s fracture principle with certain alterations so as to suit for powder metallurgy parts. Additional a statistical technique is applied to explore the value for the constants obtained in the mathematical model, which is finally used for making workability limit plot to disclose the characteristics nature of sintered Al-2TiC, Al-4TiC, Al-2WC, Al-4WC, Al-2Fe3C, Al-4Fe3C, Al-2Mo2C and Al-4Mo2C compacts under hot upsetting.

6.2 Theoretical analysis - Oyane’s fracture model
The formability stress index ($\beta$) is used to describe the effect of hydrostatic stress ($\sigma_m$) and the effective stress ($\sigma_{eff}$) on the forming limit of powder metallurgy materials during
upset forging. The formability stress index can be expressed as (Rahman & El-Sheikh 1995)
\[ \beta = \frac{3\sigma_m}{\sigma_{eff}} \]  
(6.1)

The formability stress index under triaxial stress state condition (Eq. (6.1)) can be written as
\[ \beta = 3 \left[ \frac{(\sigma_m / \sigma_z)^{1/3}}{(\sigma_{eff}/\sigma_z)^{1/3}} \right] \]  
(6.2)

Shima & Oyane (1976) proposed a fracture model as
\[ \varepsilon_{eff} \int_0^1 \left( 1 + \frac{(\sigma_m / C_1 \sigma_{eff})}{\sigma_{eff}} \right) d\varepsilon_{eff} = C_2 \]  
(6.3)

where \( C_1 \) and \( C_2 \) are material constants.

Using Eq. (6.1), Oyane’s fracture model can now be written as
\[ \varepsilon_{eff} \int_0^1 \left( 1 + \frac{\beta/3C_1}{\sigma_{eff}} \right) d\varepsilon_{eff} = C_2 \]  
(6.4)

The formability stress index at fracture is constant and Eq. (6.4) can be transformed to
\[ \varepsilon_{eff} = \frac{3C_1 C_2}{(3C_1 + \beta / 3)} \]  
(6.5)

where \( \beta / 3 \) is the value of \( \beta \) at fracture and \( \varepsilon_{eff} \) is the effective strain at fracture. Eq. (6.5) obtained from Oyane’s fracture criterion involving two constants, \( C_1 \) and \( C_2 \), describes the workability limit in the form of a hyperbola. The effective strain (\( \varepsilon_{eff} \)) in terms of cylindrical coordinates as explained elsewhere (Narayanasamy, Anandakrishnan & Pandey 2008; Doraivelu et al. 1984) under triaxial stress state condition is expressed as
\[ \varepsilon_{eff} = \left[ \frac{1}{(3/(2 + R))} \right] \left[ (\varepsilon_z - \varepsilon_{\theta})^2 + (\varepsilon_{\theta} - \varepsilon_z)^2 \right] + \left( (\varepsilon_z + 2\varepsilon_{\theta})^2 / 3 \right) \left[ (1 - R^2) \right]^{0.5} \]  
(6.6)

where \( \varepsilon_{\theta} \) is the true hoop strain and \( \varepsilon_z \) is the true axial strain.

### 6.3 Results and discussions

The upsetting tests on Al-4TiC, Al-4WC, Al-4Fe\textsubscript{3}C and Al-4Mo\textsubscript{2}C cylindrical powder metallurgy preforms were carried out. For each composition, three different aspect ratios...
(height to diameter ratio) with two different percentage theoretical densities were prepared and hot deformed without any lubricant, thus providing six different sets of experimental data for each composition. Aspect ratios of 0.2, 0.4 and 0.6 and initial theoretical density of 82% and 86% were used in this experiment. The initial height, diameter and preform density were measured and the same was measured including the bulged diameter for different strain levels until preform fracture. From the measured parameters the effective strain ($\varepsilon_{eff}^f$) and stress formability factor ($\beta^f$) at fracture were determined for Al-4TiC, Al-4WC, Al-4Fe$_3$C and Al-4Mo$_2$C as shown in Table 6.1.

Table 6.1: Effective strain and stress formability factor at fracture.

<table>
<thead>
<tr>
<th>Composition</th>
<th>Relative density</th>
<th>Aspect ratio</th>
<th>$\varepsilon_{eff}^f$</th>
<th>$\beta^f$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-4TiC</td>
<td>0.82</td>
<td>0.2</td>
<td>0.1506</td>
<td>3.4845</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.4</td>
<td>0.1820</td>
<td>2.8096</td>
</tr>
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<td></td>
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<td>0.6</td>
<td>0.1698</td>
<td>2.0257</td>
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<td>0.86</td>
<td>0.2</td>
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<td>4.3333</td>
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<td>0.1462</td>
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<td>0.6</td>
<td>0.1559</td>
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<td>Al-4WC</td>
<td>0.82</td>
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<td>0.1882</td>
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<td>Al-4Fe$_3$C</td>
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<td>0.1321</td>
<td>3.0028</td>
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<td>0.86</td>
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<td>0.4</td>
<td>0.0901</td>
<td>4.5001</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.6</td>
<td>0.1769</td>
<td>2.7961</td>
</tr>
<tr>
<td>Al-4Mo$_2$C</td>
<td>0.82</td>
<td>0.2</td>
<td>0.2938</td>
<td>2.5641</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.4</td>
<td>0.1789</td>
<td>2.6111</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.6</td>
<td>0.2161</td>
<td>2.4735</td>
</tr>
<tr>
<td></td>
<td>0.86</td>
<td>0.2</td>
<td>0.1260</td>
<td>3.5085</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.4</td>
<td>0.1664</td>
<td>2.9745</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.6</td>
<td>0.1706</td>
<td>2.8182</td>
</tr>
</tbody>
</table>
It can be seen from Table 6.1 that $\varepsilon_{\text{eff}}^f$ rises and $\beta^f$ decreases with increasing aspect ratio regardless of initial relative density and compositions. Additional, $\varepsilon_{\text{eff}}^f$ is found to be higher for lower initial relative density compacts in comparison to higher relative density compacts. In general, $\varepsilon_{\text{eff}}^f$ was found to be higher in WC reinforced aluminium matrix and lowest in Fe₃C strengthen aluminium matrix. Furthermore, $\beta^f$ is found to be higher for higher initial relative density compacts in comparison to lower relative density compacts. In general, $\beta^f$ was found to be lowest in WC reinforced aluminium matrix and highest in TiC strengthen aluminium matrix.

Using the least square method and the experimental data ($\varepsilon_{\text{eff}}^f$ & $\beta^f$) obtained for Al-4TiC, Al-4WC, Al-4Fe₃C and Al-4Mo₂C, the constants, $C_1$ and $C_2$, in the approximation to the workability limit equation (Eq. (6.5)) were determined and presented in Table 7.2 together with the approximation equations.

Table 6.2: Approximation equation and the respective constants.

<table>
<thead>
<tr>
<th>Composition</th>
<th>Constants</th>
<th>Approximation equation</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$C_1$</td>
<td>$C_2$</td>
</tr>
<tr>
<td>Al-4TiC</td>
<td>1.640</td>
<td>0.2557</td>
</tr>
<tr>
<td>Al-4WC</td>
<td>-0.0483</td>
<td>-1.9427</td>
</tr>
<tr>
<td>Al-4Fe₃C</td>
<td>0.0296</td>
<td>4.7573</td>
</tr>
<tr>
<td>Al-4Mo₂C</td>
<td>-0.4174</td>
<td>-0.2254</td>
</tr>
</tbody>
</table>

Figures 6.1-6.4 shows the experimental data’s plotted with the approximation equation given in Table 6.2 for the respective compositions, $\varepsilon_{\text{eff}}^f$ against $\beta^f$. The experimental points and the approximation to workability limit for all the composition are in good agreement and is applicable when fracture occurs at free surface for powder metallurgy materials.
The workability limit plot depicts that effective strain at fracture is a function of formability stress index at fracture \((\varepsilon_{eff} = f(\beta^f))\). The general characteristics of the plot in Figures 6.1-6.4 are similar as \(\varepsilon_{eff}^f\) decreases with the increasing \(\beta^f\). The slope of the curve is found to be highest in Mo2C composite and lowest for TiC composite depicted from Figures 6.1-6.4. These plots are important for industrial applications as safe zone and unsafe zone can be used from these plots during the forming process.

The workability limit of the TiC reinforced preforms was found to be higher followed by Fe3C and Mo2C composites. WC reinforced preforms exhibited lowest workability limit for any specified effective strain. The material compositions are designed by weight fraction and TiC particulate being the smallest density followed by Fe3C, Mo2C and WC as seen in Table 4.1. This means the amount of smaller and fine voids existing in Al-4TiC compact is more compared to other materials tested. The actual closing of voids is more in TiC compacts during hot upsetting increasing the density which intend gives high workability limit for TiC compacts.

\[
\varepsilon_{eff}^f = \frac{1.2580}{(4.920 + \beta^f)}
\]

Figure 6.1: Workability limit for Al-4TiC.
Figure 6.2: Workability limit for Al-4WC.

Figure 6.3: Workability limit for Al-4Fe₃C.
Figure 6.4: Workability limit for Al-4Mo$_2$C.

Similar plots are drawn for Al-2TiC, Al-2WC, Al-2Fe$_3$C and Al-2Mo$_2$C to further confirm the validity of the method and approximation techniques. It is seen that the experimental values obtained for the $\beta'$ and the $\varepsilon'_{\text{eff}}$  is in good agreement with the approximation equations determined by the Oyane’s fracture criteria. The constants $C_1$ and $C_2$ are given in Table 6.3 with plots in Figures 6.5-6.8 of the Oyane’s fracture approximation equations and the experimental values. These workability limit diagrams can be used effectively in the forming process of such materials. The failure zones can be identified from these plots and can be used in the designing of the forging operation and the dies so as to stop the pressing operations before failure or employ the re-pressing activity on the unconstrained surfaces before failure.

Table 6.3: Approximation equation.

<table>
<thead>
<tr>
<th>Composition</th>
<th>$C_1$</th>
<th>$C_2$</th>
<th>Approximation equation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al-2TiC</td>
<td>1.2351</td>
<td>0.3584</td>
<td>$\varepsilon'_{\text{eff}} = 1.3280 / (3.7053 + \beta')$</td>
</tr>
<tr>
<td>Al-2WC</td>
<td>-0.3584</td>
<td>-0.2648</td>
<td>$\varepsilon'_{\text{eff}} = 0.2847 / (-1.0752 + \beta')$</td>
</tr>
<tr>
<td>Al-2Fe$_3$C</td>
<td>-0.0029</td>
<td>-58.4200</td>
<td>$\varepsilon'_{\text{eff}} = 0.5083 / (-0.0087 + \beta')$</td>
</tr>
<tr>
<td>Al-2Mo$_2$C</td>
<td>-0.4017</td>
<td>-0.2972</td>
<td>$\varepsilon'_{\text{eff}} = 0.3582 / (-1.2051 + \beta')$</td>
</tr>
</tbody>
</table>
Figure 6.5: Workability limit for Al-2TiC.

\[ \varepsilon_{\text{eff}} = 1.3280/\left(3.7053 + \beta'\right) \]

Figure 6.6: Workability limit for Al-2Fe₃C.

\[ \varepsilon_{\text{eff}} = 0.5083/\left(-0.0087 + \beta'\right) \]
6.4 Conclusions

The theoretical correlation between effective strain and formability stress factor at fracture was acquired, which was used for constructing workability limit plot for Al-2TiC, Al-2WC, Al-2Fe₃C, Al-2Mo₂C, Al-4TiC, Al-4WC, Al-4Fe₃C and Al-4Mo₂C. It is established that hyperbolic curve acquired from Oyane’s fracture criterion for the theoretical relationship showed good agreement with the experimental results; hence these workability limit plots can be used in forming processes.
Chapter 7
Studies on Formability of Sintered Aluminum Composites during Hot Deformation using Strain Hardening Parameters

7.1 Overview
The forming limit of powder metallurgy compacts is absolute important and the study of workability behavior of powder metallurgy materials is an essential study in designing of the deformation process (Zhang et al. 2009; Ramesh & Senthivelan 2010). Workability of the powder metallurgy compacts plays a key part to find out if the powder metallurgy compact is shaped effectively or fracture starts during the deformation process. Many researchers (Narayan & Rajeshkannan 2011; Rajeshkannan 2010; Butuc, Gracio & Rocha 2006) have worked and evaluated the forming limit of powder metallurgy materials and used the analysis in preform shape design and die constraints designs. These are important as to arrest cracks before it appears on the free surface during the forming process.

The present investigation proposes to study the workability behavior by studying the strain hardening parameters, strain hardening exponent ($n_i$) and stress coefficient ($K_i$). Several investigators studied strain hardening behavior to evaluate the densification and strain hardening behavior, however, hardly any research is conducted to find the forming limit using the two very important strain hardening parameters. The strain hardening coefficient and the stress coefficient are one of the basic forming constraints of metal matrix composites. Strain hardening studies are vital in the deformation process as it governs the extent of even plastic strain the metal can sustain throughout the deformation without failure.

Narayanasamy, Ramesh and Pandey (2005) explored on the instant strain-hardening performance of a powder metallurgy aluminum-iron alloy. The effect of various iron content and preform geometry on strain hardening and densification were established. Luo et al. (2010) studied the influence of temperature, strain rate and strain on strain hardening. Further, a correlation between strain hardening parameters ($n_i$ and $K_i$) with
axial strain and relative density was developed experimentally and used to evaluate the geometric and matrix work hardening (Rajeshkannan & Narayan 2009, 2013). An interesting point to note from these researches (Luo et al. 2010; Rajeshkannan & Narayan 2009, 2013) is that during the initial stages the strain hardening increases rapidly and then decreases sharply. This behavior is due to large degree of deformation during the initial stages of deformation and needs to be neglected for all practical reasons. Then the strain hardening values maintain a steady behavior and finally during the last phase of forming there is fluctuation in the strain hardening values. This final stage can be analyzed to plot the forming limit diagram and determine the failure zone. The sintered forging process is widely used in industries for producing parts with uniform properties and complex geometry. Further, one of the quests today is producing high strength materials using green manufacturing such as powder metallurgy manufacturing process.

The chapter analyzes the formability limit of powder metallurgy preforms of Al-2TiC, Al-2WC, Al-2Fe₃C, Al-2Mo₂C, Al-4TiC, Al-4WC, Al-4Fe₃C and Al-4Mo₂C experimentally with the influence of preform geometry, initial relative density and various carbide reinforcements. The instantaneous strain hardening (ni) and instantaneous stress coefficient (Ki) is used to plot the forming limit diagram.

7.2 Theoretical analysis - fracture model using Ludwik’s equation

Doraivelu et al. (1984), Shima & Oyane (1976), Park (1995), and Lee & kim (1992) studied the yield criterion parameters for Eq. (4.6) as presented in Table 7.1. It can be seen in Table 7.2 that Doraivelu et al. (1984), Park (1995) and Lee & kim (1992) have the same values for the yield criterion parameters, $A$ and $B$, however, all have different values for yield criterion parameter, $\delta$. To highlight the modifications, the values for $\delta$ is calculate for different relative density of sintered powder metallurgy parts, as given in Table 7.2. The range of relative density for powder metallurgy materials is in the range of 0.80 to 1.0 for many industrial applications as well as for research. Hence, the initial density chosen for analysis in Table 7.2 is 0.80 and the relative density is varied in a range of 0.80 to 1.0 in increments of 0.04. It can be seen that when the relative density approaches 1, that is when the powder metallurgy material is well compacted to
approximately fully dense material, the values of \( \delta \) approach to 1.0 and are in close proximity with each other except for Lee & kim (1992). The difference in the values of \( \delta \) for Lee & kim (1992) with other researchers is remarkable. Further, it can be seen that for relative density greater than 0.80, \( \delta \) is positive and the square of yield strength, \( Y \), in Eq. (4.6) will be positive and does not violate the yield state of uniaxial and triaxial compression. Moreover, at the apparent relative density, \( R = 0.3 \), the \( \delta \) is less than zero indicating that the loose powder have zero strength. Shima & Oyane (1976) have all yield criterion parameters different from other researchers as presented in Table 7.1, however, their values for \( A \) and \( B \) are same with all other researchers as can be seen in Table 7.2 while the values for \( \delta \) is in close range of Doraivelu et al. (1984) and Park (1995).

Table 7.1: Yield criterion parameters

<table>
<thead>
<tr>
<th>Researcher</th>
<th>( A )</th>
<th>( B )</th>
<th>( \delta )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Doraivelu et al. (1984)</td>
<td>( 2 + R^2 )</td>
<td>( (1 - R^2)/3 )</td>
<td>( 2R^2 - 1 )</td>
</tr>
<tr>
<td>Shima &amp; Oyane (1976)</td>
<td>( 3/(1+0.6889(1 - R)^{1.028}) )</td>
<td>( 0.6889(1 - R)^{1.028} )</td>
<td>( R^5/(1+0.6889(1 - R)^{1.028}) )</td>
</tr>
<tr>
<td>Park (1995)</td>
<td>( 2 + R^2 )</td>
<td>( (1 - R^2)/3 )</td>
<td>( 1.44R^5/(2.44 - R) )</td>
</tr>
<tr>
<td>Lee &amp; Kim (1992)</td>
<td>( 2 + R^2 )</td>
<td>( (1 - R^2)/3 )</td>
<td>( ((R – R_c)/(1 – R_c))^2 )</td>
</tr>
</tbody>
</table>

Table 7.2: Relationship between the constant \( A \), \( B \) and \( \delta \) and the relative density.

<table>
<thead>
<tr>
<th>( R )</th>
<th>0.84</th>
<th>0.88</th>
<th>0.92</th>
<th>0.96</th>
<th>1</th>
</tr>
</thead>
<tbody>
<tr>
<td>( A )</td>
<td>( B )</td>
<td>( \delta )</td>
<td>( A )</td>
<td>( B )</td>
<td>( \delta )</td>
</tr>
<tr>
<td>Doraivelu et al. (1984)</td>
<td>2.71</td>
<td>0.10</td>
<td>0.41</td>
<td>2.77</td>
<td>0.08</td>
</tr>
<tr>
<td>Shima &amp; Oyane (1976)</td>
<td>2.72</td>
<td>0.10</td>
<td>0.38</td>
<td>2.78</td>
<td>0.08</td>
</tr>
<tr>
<td>Park (1995)</td>
<td>2.71</td>
<td>0.10</td>
<td>0.38</td>
<td>2.77</td>
<td>0.08</td>
</tr>
<tr>
<td>Lee &amp; Kim (1992)</td>
<td>2.71</td>
<td>0.10</td>
<td>0.04</td>
<td>2.77</td>
<td>0.08</td>
</tr>
</tbody>
</table>

---

117
As explained above, the following yield criteria parameters are chosen to calculate the effective or equivalent stress in the powder metallurgy materials as \( A = 2 + R^2 \), \( B = (1 - R^2) / 3 \), \( \delta = 2R^2 - 1 \). Eq. (4.11) can now be written as

\[
Y_o = \sigma_{eff} = \left[ \frac{(\sigma_r^2 + 2\sigma_\theta^2 - R^2(\sigma_\theta^2 + 2\sigma_\theta\sigma_z))}{2R^2 - 1} \right]^{0.5}
\]  
(7.1)

Mean stress under the assumption that \( \sigma_\theta = \sigma_r \) for cylindrical coordinate system is:

\[
\sigma_m = \frac{\sigma_r + \sigma_\theta + \sigma_z}{3} = \frac{2\sigma_\theta + \sigma_z}{3}
\]  
(7.2)

The stress formability factor (\( \beta \)) demonstrates the impact of hydrostatic and equivalent stress on the forming limit and can be expressed as (Narayanasamy, Ponalagusamy & Subramanian 2001)

\[
\beta = \frac{3\sigma_m}{\sigma_{eff}}
\]  
(7.3)

The effective strain (\( \varepsilon_{eff} \)) in cylindrical axes (Narayanasamy, Ramesh & Pandey 2005) is expressed as:

\[
\varepsilon_{eff} = \left[ \frac{2}{(3(2 + R))} \left[ (\varepsilon_z - \varepsilon_\theta)^2 + (\varepsilon_\theta - \varepsilon_z)^2 \right] + \left( (\varepsilon_z + 2\varepsilon_\theta)^2 / 3 \right)(1 - R^2) \right]^{0.5}
\]  
(7.4)

where \( \varepsilon_\theta = \) hoop strain and \( \varepsilon_z = \) axial strain and is given as:

\[
\sigma_z = \frac{\text{load}}{\text{contact surface area}}
\]  
(7.5)

\[
\varepsilon_z = \ln \left( \frac{h_f}{h_o} \right)
\]  
(7.6)

\[
\varepsilon_\theta = \varepsilon_r = \ln \left( \frac{D_f}{D_o} \right)
\]  
(8.7)

where \( h_o = \) initial height; \( h_f = \) final height; \( D_o = \) initial diameter and \( D_f = \) final contact diameter.

Further, Ramesh and Senthivelan (2010) considered the forged diameters in determining the hoop strain stated as below

\[
\varepsilon_\theta = \ln \left[ \frac{2D_b^2 + D_f^2}{3D_o^2} \right]
\]  
(7.8)
where \( D_b = \) final bulged diameter and \( D_c = \) final contact diameter.

Equation (4.6) can be written using the Ludwik equation as:

\[
AJ'_2 + BJ'_1 = Y^2 = \delta(K \epsilon_{\text{eff}}^n)^2
\]

where \( K = \) stress coefficient and \( n = \) hardening exponent. Equations (7.1) and (7.4) is used to evaluate the stress coefficient and the hardening exponent in the modified Ludwik equation for powder metallurgy materials to evaluate the instantaneous stress coefficient \((K_i)\) and instantaneous hardening exponent \((n_i)\), where \( \sigma \) is effective stress and \( \epsilon \) is equivalent strain. The derivation is as follows:

Taking successive stress on the compact as 1, 2, 3, . . . , (j-1) and j. Thus, the Ludwik equation gives:

\[
\sigma_j = K \epsilon_j^n
\]

\[
\sigma_{j-1} = K \epsilon_{j-1}^n
\]

Deducting Eqs. (7.10) and (7.11) gives

\[
K_i = \frac{\sigma_j - \sigma_{j-1}}{\epsilon_j^n - \epsilon_{j-1}^n}
\]

Now, dividing the Eq. (7.10) by Eq. (7.11) and taking the natural logarithm gives

\[
n_j = \frac{\ln \left( \frac{\sigma_j}{\sigma_{j-1}} \right)}{\ln \left( \frac{\epsilon_j}{\epsilon_{j-1}} \right)}
\]

Equations (7.12) and (7.13) are used to find the stress coefficient and hardening exponent, respectively.

### 7.3 Results and discussions

During the deformation of powder metallurgy parts, it is known (Narayanasamy & Selvakumam 2005; Rajeshkannan et al. 2008; Kuhn & Lawley 1978; Rajeshkannan, Pandey & Shanmugam 2008; Lewis & Khoei 2001) that density is constantly improved due to induced strain. Figure 7.1 gives the correlation of relative density and axial strain, \( \epsilon_z \) showing the densification characteristics of sintered aluminium composites for
varying initial densities and different carbide reinforcement. The induced hydrostatic stress present in the preforms during the upsetting operation has a major part in pore elimination of powder metallurgy parts and depends on friction, porosity, geometry and respective compositions. The extent of densification is not the same for varying initial densities and different carbide reinforcement. Higher values of densification rate are observed for smaller initial density preforms. The reason for this is the higher requirements of hydrostatic force to close more number of pores found in the lower initial relative density compacts resulting in large amount of densification. The same can be verified from Fig. 7.2. Al-4TiC composite exhibited greater densification rate and greater final density followed by Al-4Fe₃C composite and then Al-4Mo₂C composite. Al-4WC composite showed smallest densification rate and smallest final density attainment. Further, the densification rate during the final stages of deformation is notably lower when compared to the initial stage. During the forming practice the particles and pores stretch perpendicular to the load axis (Fig. 7.3). This provides more material resistance to deformation. The shapes of pores after sintering process are nearly spherical (Fig. 7.3a) while during the later stages of deformation it becomes almost cylindrical in shape (Fig. 7.3b).

Workability limit diagram is used as a reference to estimate the failure during metal forming and can be used effectively for powder metallurgy forming as complex deformation mechanics is involved. The workability limit diagram shows the safe zone and failure zone in further processing of the powder metallurgy products and can be used effective to understand when to activate re-pressing process on the free surface of powder metallurgy part to avoid cracks and failure. This will help in the design of dies and process operation for powder metallurgy close die forging. Once the pores penetrates to the surface of the compact, it is difficult to repair the crack by re-pressing or other process and hence, this workability limit diagram is important in the forming of powder metallurgy parts and in die design.

It is well understood that these two parameters, strain hardening exponent and stress coefficient, are important to study in the forming of MMC’s. Several researchers have
used these two parameters in the development of successful powder metallurgy parts and procedures (Narayanasamy, Ramesh & Pandey 2005; Selvakumar & Narayanasamy 2003; Straffelini 2005; Ni et al. 2014). Further, Narayan and Rajeshkannan (2011) introduced two new parameters called density hardening exponent and density strength coefficient to study the hardening phenomenon of sintered plain carbon steel preforms. Figures 7.4-7.7 shows the workability limit diagram plotted using instantaneous $ni$ and instantaneous $Ki$ against relative density. The initial aspect ratio is chosen to be 0.4. The $ni$ and $Ki$ is calculated using the equations derived from the logarithmic graph of axial stress and strain as explained in detail in section 7.2. At the start of deformation the applied stress is significant in comparison to the axial deformation as to overcome the initial yield stress. Initially the applied stress are not adequate to collapse the large amount of pores and the stress values rise considerably for small amounts of densification and deformation resulting in higher $ni$ values initially. This does not indicate strain hardening in the material and is always ignored (Narayan & Rajeshkannan 2011). Hence, Figures 7.4-7.7 are plotted for intermediate and final stages of deformation.

In all the plots the safe zone, failure zone and critical relative density is shown. The zones and critical relative density is determined by the changeover point on the slope of the linear line which defines the minimum relative density of the sintered parts before existing pores may appear on the free surface of the parts causing the failure. Workability is the amount to which the material can be deformed before failure. In the final stages of deformation the powder metallurgy part nears full density with 5 to 8% porosity, which is hard to eliminate. The powder metallurgy parts are extremely strain hardened at this stage, hence, to further eliminate the residual pores require high load that considerably increases the strain hardening values and when these remaining pores collapse it considerably decreases the strain hardening values. This causes the instabilities in the $ni$ and $Ki$ values during the final stages of deformation. The same is also presented by Narayan and Rajeshkannan (2011) and Rajeshkannan et al. (2008). These fluctuations in the $ni$ and $Ki$ values during the last stage of forming is a sign of failure if deformation is not stopped or if re-pressing on the free surface in close die forging is not employed. The
critical density can be used in die design and to produce defect free parts. For any axial strain, the hydrostatic stress is higher for Al-4TiC composites, followed by Al-4Fe$_3$C, Al-4Mo$_2$C and smallest for Al-4WC composites as seen in Fig. 7.2. This means Al-4WC preforms has room for further deformation as long as it has no visible cracks on the free surface. The workability diagram of Al-4WC (Fig. 7.7) can be effectively utilized here for this purpose.

In Figures 7.4-7.7 the critical relative density is found to be 93.5%, 90.8%, 89.2% and 87.3% for powder metallurgy Al-4TiC, Al-4Fe$_3$C, Al-4Mo$_2$C and Al-4WC, respectively. These critical relative densities give the workability of the respective materials to produce defect free parts. Once the respective critical relative density is reached during deformation the chances of cracks appearing is high and hence deformation needs to stop or repressing needs to be employed to produce healthy powder metallurgy aforementioned parts. It is noted that the critical relative density is highest for TiC reinforced aluminium, followed by Al-4Fe$_3$C composite and then Al-4Mo$_2$C composite. Al-4WC composite had the smallest critical relative density. One of the reasons for this is that the formability of TiC reinforced aluminium composites was found to be higher followed by Fe$_3$C and Mo$_2$C reinforced aluminium composites. WC reinforced aluminium composites gave the smallest formability stress index against axial strain (Fig. 7.8). Further, the composites are designed by weight ratio and titanium carbide particles had the lowest weight followed by iron carbide, molybdenum carbide and then tungsten carbide (Table 4.1). Hence, the amount of smaller and fine pores existing in Al-4TiC compact is higher compared to other materials tested here and hence, for the same reason the critical relative density is higher for TiC composite in comparison with other composites.
Figure 7.1: Correlation between relative density and axial strain for initial relative density of 0.86.

Figure 7.2: Correlation between mean stress, $\sigma_m / \sigma_{eff}$ and axial strain for initial relative density of 0.86.
Figure 7.3: Micrographs of Al-4TiC at the edge of the specimen: a) undeformed and b) fully deformed.

Figure 7.4: Workability limit diagram for powder metallurgy Al-4TiC composites.
Figure 7.5: Workability limit diagram for powder metallurgy Al-4Fe$_3$C composites.

Figure 7.6: Workability limit diagram for powder metallurgy Al-4Mo$_2$C composites.
Figure 7.7: Workability limit diagram for powder metallurgy Al-4WC composites.

Figure 7.8: Correlation between formability stress index, $\beta$ and axial strain for initial relative density of 0.86.
7.4 Conclusion

The workability behavior of Al-2TiC, Al-2WC, Al-2Fe₃C, Al-2Mo₂C, Al-4TiC, Al-4WC, Al-4Fe₃C and Al-4Mo₂C are analyzed and the finding are as follows,

- The amount of densification is higher in TiC reinforced aluminium followed by Fe₃C, then Mo₂C and lowest for WC reinforced aluminium composites. Further, smaller aspect ratio composites gave good densification rate than bigger aspect ratio composites. Higher values of densification rate are observed for smaller initial relative density preforms. This ensured better formability behavior for TiC reinforced aluminium compacts and smaller aspect ratio compacts. The accompanying compressive hydrostatic stress is responsible for densification.

- The particles and porosity deform and elongate perpendicular to the direction of load application.

- The instantaneous strain hardening exponent and instantaneous stress coefficient are utilized to plot the forming limit diagrams highlighting the safe working zones. These plots can be effectively used in the design of forming operations for the aforementioned composites.

- The critical relative density is found to be 93.5%, 90.8%, 89.2% and 87.3% for powder metallurgy Al-4TiC, Al-4Fe₃C, Al-4Mo₂C and Al-4WC, respectively. The safe working zone is found to be the narrowest for Al-4WC.

- The critical relative density is found to be 93.5%, 90.8%, 89.2% and 87.3% for powder metallurgy Al-2TiC, Al-2Fe₃C, Al-2Mo₂C and Al-2WC, respectively. The safe working zone is found to be the narrowest for Al-2WC.
Chapter 8
Corrosion behavior of sinter forged aluminum composites during hot deformation

8.1 Overview
The presence of pores in the powder metallurgy parts especially open ones has significant effect on the corrosion resistance of sinter-forged aluminium composites. Wear and corrosion are the main reasons of powder metallurgy products failure in industry and these limit its application to corrosive and wear free environments. Solving these wear and corrosion problems will enhance the potential use of powder metallurgy parts for many industrial applications (Gabe 1977; Nash 1990).

Jinsun, Hotta and Mori (2012) presented improved resistance of high strength Mg-Al-Mn-Ca magnesium alloy made by powder metallurgy process. They found that the SAWP process greatly improved the corrosion resistance due to dispersion of intermetallic phase. An emersion test method was used for the corrosion study. Dobrzanski, Wlodarczyk and Adamiak (2005) conducted corrosion test using the measurement system consisting of the PG P-21 potentiostat working with the radiometer Copenhagen voltmeter software. The corrosion behavior of varying composites was varying as well as the pitting amount and place on the exposed surface. Conventional electrochemical potentio-dynamics tests used by Marchiori et al. (2007) in both cathode configuration and anode configuration to evaluate corrosion of plasma sintered unalloyed iron. Neubert et al. (2007) studied mechanical properties and corrosion behavior of Al-Sc-Zr alloy prepared by powder metallurgy and reported the corrosion resistance and mechanical properties of Al-Sc-Zr is better than AA6061-T6 Al alloy. This was due to phase precipitation of Al$_3$ (Sc, Zr) particles during hot extrusion. Again they have used immersion test for 6 hours to determine the corrosion rate. Mamatha, Pruthviraj and Ashok (2011) presented corrosion behavior of aluminium metal matrix composites reinforced with SiC particulates in HCl solution using weight loss method for 96 hours. The effect of varying SiC particulates in the aluminium composites on corrosion rate is evident. The corrosion resistance of iron powder metallurgy parts can be improved by
surface treatment, nickel plating, coating, cathodic and anodic protection (Ettaat et al 2009; Leisner, Leu & Moller 1997). Steam treatment of sintered ferrous parts at 500°C improves the hardness, wear and corrosion properties on the exposed surface due to the formation of thin Fe$_3$O$_4$ layer on the exposed surface (Wick & Veilleux 1985; Beiss 1991). The effect of WC content on the microstructure and corrosion behavior of Ti (C, N) based cermet’s in nitric acid solution was studied by Chenghong et al. (2013). It was observed that WC is more easily oxidized and dissolved in nitric acid solution compared to Ti (C, N), hence, the corrosion rate of cermet’s increases and the corrosion resistance of Ti (C, N) based cermet’s decreases with increasing WC content.

The standard technique used for evaluating corrosion tests of powder metallurgy parts is immersion weight loss method (Chandramouli et al. 2007; Lei, Maicang & Jianxin 2011; Kumar, Rao & Girish 2012). Further, some of the immersion solutions used by several researchers are 3.5wt% NaCl solution, 1 M HCl solution, 9wt% HNO$_3$ solution, 0.5 M KNO$_3$ solution; however, the use of immersion solution is consistent throughout the whole experiment (Alaneme & Bodunrin 2011; Bobic et al. 2009; Sharma 2001). There are many non-destructive techniques (NDT) used such as linear polarization resistance (LPR) method (Scully 2000), galvanostatic pulse technique (Deo, Birbilis & Cull 2014), potentio-dynamic polarization test (Jinsun, Hotta & Mori 2012) and electrochemical impedance spectroscopy (Sherif et al. 2011).

Currently no electrochemical process is well defined to measure the corrosion dynamics of aluminium metal matrix composites. Birbilis, Nairn and Forsyth (2003) presented the problems encountered whilst using LPR method to study steel corrosion in concrete samples. They introduced a new technique to overcome problems encountered by other electrochemical process; the galvanostatic pulse technique. In this technique short galvanostatic pulse is used moving away from other technique using longer pulses with greater error (Lu & Peiyu 2000). Deo, Birbilis and Cull (2014) have successfully used this galvanostatic pulse technique successfully to study metal corrosion in different soils.
Based on the published literature, there is less information about the corrosion behavior of aluminium metal matrix composite with the effect of WC, Mo$_2$C and Fe$_3$C reinforcements. In the present study a galvanostatic pulse technique is used to evaluate the corrosion behavior of several aluminium metal matrix composites such as Al-1TiC, Al-2TiC, Al-3TiC, Al-4TiC, Al-2Fe$_3$C, Al-4Fe$_3$C, Al-2Mo$_2$C, Al-4Mo$_2$C, Al-2WC and Al-4WC. Further, the effect of initial porosity levels in aforementioned composites on corrosion resistance was also investigated and presented in this chapter. A very similar method was used by Jinsun, Hotta and Mori (2012) to study the corrosion behavior of magnesium alloy made by rapid solidification powder metallurgy process. They have used a typical three electrode cell with a saturated Ag/AgCl reference, Pt counter and the specimen as the working electrode and same is used in our research work.

### 8.2 Aluminium composite/NaCl solution interface

An interface is formed when the aluminium composite comes in contact with the 3.5% NaCl water solution. This interface can be modelled by an equivalent circuit called the Randle’s circuit. The circuit reassembles $R_P C_{dl}$ parallel arrangement in series with NaCl solution resistance, $R_\Omega$, whereby $R_P$ is the polarization resistance and $C_{dl}$ is the double layer capacitance shown in Fig. 8.1.a. The capacitance associated with the aluminium composite will vary depending on the reinforcements present in the composites. The double layer capacitance is non-ideal due to the $\beta_C$-parameter. $\beta_C$-parameter is an indicator of dispersion characteristics. It can be used to illustrate how the surface is taking part in the corrosion process. When $\beta_C = 1$, it indicates ideal uniform corrosion and $C_{dl}$ is an ideal capacitor. When $\beta_C < 1$, it indicates localized corrosion and $C_{dl}$ is a non-ideal capacitor.
8.3 Experimental Details

8.3.1 Specimen preparation

The materials used in the current investigation are aluminium powder of 150 μm and titanium carbide, tungsten carbide, molybdenum carbide and iron carbide, of 50 μm. All powders used in the present investigation had purity levels of 99.7%.

The required amount of powders was mixed to obtain the aforementioned aluminium carbide composites using planetary ball milling machine. The ball mill was run for 2-2.5 hours at 200 rpm to get a homogenized mixture. The apparent density was continuously measured to ensure uniform distribution of the reinforcement particles in the matrix.

The powders corresponding to cylindrical specimens with diameter of 24 mm and height of 10 mm were compacted in a pressure range of 139 MPa to 159 MPa (hydraulic press) to obtain relative density of 0.86 ± 0.01 and 0.82 ± 0.01. The specimens were coated with Al₂O₃ mixed with acetone to avoid oxidation during the sintering process. The specimens were left for atmospheric drying for a period of 24 hours.

Then the specimens were sintered in an electric muffle furnace at 220°C for 30 minutes and then at 594°C for an additional period of 60 minutes. Each specimen was compressively deformed at a temperature of 594°C to two different height strains. Finally the specimens were left for atmospheric cooling.
8.3.2 Solution Preparation
3.5% NaCl was prepared using analytical grade reagent and distilled water. Each specimen was immersed in 500 mls NaCl solution separately such that the ratio of exposed specimen surface area (cm$^2$) to volume of the NaCl solution (ml) was about 1:500.

8.3.3 Microstructure
The specimens for microstructure characterization were cut at the center with the observation plane parallel to the compression direction. The microstructures were examined using an optical microscope. The specimens were ground with 1200 grit emery and polished with 9 microns diamond paste, then with 3 microns diamond paste and finally with colloidal silica. Then the specimens were cleaned with distilled water and absolute ethanol and dried with warm flowing air. These specimens were then etched with Keller’s reagent. These were then cleaned with distilled water then ethanol and dried with warm flowing air before microstructure examination.

To study the corrosion behavior at the initial stage, another set of specimens were ground with 1200 grit emery and polished with 9 microns diamond paste, then with 3 microns diamond paste and finally with colloidal silica. These specimens were then immersed in the 3.5% NaCl water solution for 4 hours and the surface was observed with an optical microscope. The specimens were cleaned with distilled water, then with absolute ethanol and dried with warm flowing air.

8.3.4 Electrochemical measurement
A classical three electrode configuration was used with an Ag/AgCl reference electrode, platinum as counter electrode and the specimen as working electrode. All the specimens were machined to form cylinder of diameter of 1.13 cm. The specimens were then molded in a thermoplastic material to only expose a surface area of 1 cm$^2$. The exposed surface of the specimens for the test were ground using 1200 grit emery paper and cleaned with distilled water and absolute ethanol. A 2 mm hole was made into the thermoplastic material and a copper wire was inserted to just touch the specimen to allow.
galvanostatic pulse current flow. The hole was sealed with adhesive to hold the copper wire in place and to avoid the solution from entering. It was ensured that the exposed surface is not damaged. The Eco-chemie microAUTOLAB III potentiostat/galvanostat together with the General Purpose Electrochemical Software was used to supply short (1s) galvanostatic pulse and the resulting potential-time response was recorded internally by this set-up. The measurements were taken after 2 hours of immersion in 3.5% NaCl solution using a purpose-built inversion program. The counter and reference electrode were secured properly allowing contact with the solution. The depth of the immersion was kept constant. A potential of 1.5 V was supplied between the working and counter electrode for all the measurements. The potential circuit data’s were then used to plot the charging and discharging curves along with the curve-fitting techniques to extract the corrosion related parameters. A purpose-built inversion program was also used for this analysis. The following parameters were obtained such as corrosion potential (Ecorr), $\beta_C$-parameter, polarization resistance ($R_p$) double layer capacitance ($C_{dl}$) and corrosion current density ($I_{corr}$) using the software program. A schematic of the experimental setup is given in Fig. 8.2.

Figure 8.2: Illustration of the electrochemical setup in this study.
8.4 Results and Discussion

8.4.1 Microstructure

To study the corrosion behavior at the initial stage, all specimens after the standard preparation technique as discussed in section 8.3 of this chapter were immersed in 3.5% NaCl water solution for 4 hours and the surface was observed with an optical microscope. The specimens were cleaned with distilled water, then with absolute ethanol and dried with warm flowing air before microstructure viewing. Figure 8.3 gives the microstructural view of TiC reinforced aluminium and Figure 8.4 gives the micrographs of WC, Fe₃C and Mo₂C reinforced aluminium metal matrix composites. Apart from the varying carbide and varying carbide concentrations, initial relative density and final height strain is varied. A low height strain here is referred to as low deformed specimen and a higher height strain is referred to as a highly deformed specimen. Generally it was observed in most composites that lower initial relative density specimens showed poor materials resistance to corrosion. Further, low deformed specimens also showed poor materials resistance to corrosion. Specimens of 0.86 initial relative density and highly deformed specimens showed lower corrosion when compared to lower initial relative density and low deformed specimens. The presence of pores may act as the influencing factor here and lower initial relative density and low deformed specimens has higher number of pores present in the bulk material promoting corrosion. It is seen from the microstructures that corrosion is higher around the pores and also around the carbide reinforcement particles. The chemical reaction for corrosion is enhanced at the grain boundaries of two different materials, that is, respective carbides and aluminium. Further, from Figure 8.3 it can be seen that when the TiC content is increased in the specimens the surface corrosion is reduced significantly, however, spot/localized corrosion is seen and is mainly around the reinforced particles. When the TiC particles are low the corrosion activity is mainly concentrated on the whole surface area. However, as the TiC particles increase the number of weaker grain boundaries is higher, between reinforced particle and aluminium particles, and the corrosion activity shifts to these weaker grain boundaries, promoting spot/localized corrosion. Also, pitting corrosion is enhanced in the highly deformed specimens as the TiC particles increases in the specimens. This is due to
the surface variation due to presence of carbide reinforcements in composites can promote increased pitting corrosion.

From Figure 8.4 it can be stated that Mo$_2$C reinforced aluminium composites showed highest corrosion irrespective of initial relative density, percentage carbide concentrations, or high or low deformation of the specimen compared to other composites. Extensive pitting corrosion is shown by these specimens. After Mo$_2$C reinforced aluminium composites, TiC reinforced aluminium composites showed poor resistance to corrosion. Further, WC reinforced aluminium composite and Fe$_3$C reinforced aluminium composite showed better material resistance to corrosion when compared to Mo$_2$C and TiC reinforced aluminium composites. It can be concretely noted that Mo$_2$C reinforced composites prepared by primary powder metallurgy manufacturing process has very poor material resistance to corrosion. Hence, parts prepared from this combination and process needs to be well protected to avoid corrosion in actual application. Pitting corrosion starts with the formation of pits. Pit initiation starts with the absorption of Cl$^-$ ions and its chemical reaction with the oxide film (Zuhair & Amro 2002). This phenomenon is high in Mo$_2$C reinforced aluminium composites. Thus many corrosion pits exists damaging the hard particle bonds. This will lead to the falling of unsupported hard phase particles from the surface of the specimen promoting rapid corrosion.
Figure 8.3: Micrographs of powder metallurgy TiC reinforced aluminium composites after immersion of 4 hours in 3.5% NaCl.
Figure 8.4: Micrographs of powder metallurgy WC, Fe3C, Mo2C reinforced aluminium composites after immersion of 4 hours in 3.5%NaCl.
8.4.2. Electrochemical measurements

Results from short galvanostatic pulse measurements for different carbide reinforced aluminium are described here. Measurements were conducted at 2 hours after immersion in 3.5% NaCl water solution. The galvanostatic pulse technique measures the following parameters, polarization resistance, $R_p$, double layer capacitance, $C_{dl}$, pitting corrosion intensity or beta parameter, $\beta_C$, and corrosion potential, $E_{corr}$. The following parameters can be successfully used to access the corrosion behavior of the metals and calculate the corrosion rate. A simple electric circuit was implemented and the potential time response was measured after application of a short (1s) galvanostatic pulse. The details of this circuit are discussed in Deo (2013). A typical potential time response measured in this work is shown in Figure 8.5. It has two potential-time response period, first is the charging curve and the other is the discharging curve. The charging curve is modelled by a Randle’s type circuit shown in Fig. 8.1.a.

![Figure 8.5: Potential time response of Al-1TiC, charging curve with best fit curve.](image)

The corrosion related parameters ($\beta_C$, $C_{dl}$, $R_p$, $E_{corr}$) and $R_\Omega$ can be determined either by analyzing the charging curve or the discharging curve. A purpose-built inversion program is used for the measurements and analysis of results for this work and the details of the program is discussed elsewhere (Birbilis & Cherry 2005). An excellent feature of the purpose-built inversion program is that the corrosion parameters can be determined by
just analyzing the charging curve without the need of the discharging curve. On the other hand, if discharging curve is used to obtain the results then the charging curve needs to be analyzed first as the discharging curve analysis requires some input from the charging curve. Hence, as we just need the corrosion parameters ($\beta_C$, $C_{dl}$, $R_p$, $E_{corr}$) only the charging curve is used for this work, the best curve fitting techniques is only applied to the charging curve using the purpose-built program with error bars.

An increase in polarization resistance, $R_p$, values and a decrease in double layer capacitance values, $C_{dl}$, indicate an increase in materials resistance to corrosion. Similar method and analysis is discussed by Birbilis and Cherry (2005) to evaluate corrosion behavior of reinforced steels in concrete. Further, as the polarization resistance, $R_p$, increases the corrosion current decreases indicating low corrosion. A higher $C_{dl}$ value indicates greater ability to store electric charge by the working electrode. This will create higher corrosion density indicating increased corrosion. It is noted that the distance between the reference electrode and the working electrode for all experimental work in this research is kept constant. Further, the test solution is also kept constant for all experimental works. Hence the $R_p$ and $C_{dl}$ values only depends on the variables tested in this research work which are carbide concentrations, height strain and initial relative density. Figure 8.6 gives the variation of $C_{dl}$ against $R_p$. A strong inverse relationship between $R_p$ and $C_{dl}$ in log-log plot is depicted from Figure 8.6. Hence, $R_p$ and $C_{dl}$ corrosion parameters can be effectively used to study the corrosion behavior. This galvanostatic pulse corrosion parameters, $\beta_C$, $C_{dl}$, $R_p$, is effectively used to study reinforced steel corrosion in concrete slabs (Birbilis, Nairn & Forsyth 2003; Lu & Peiyu 2000; Zuhair & Amro 2002; Deo 2013; Birbilis & Cherry 2005). The authors have effectively utilized $\beta_C$, $C_{dl}$, $R_p$ parameters to study corrosion behavior.
Figure 8.6: Variation between polarization resistance and double layer capacitance for different composites measured at 2 hours of immersion.

Figure 8.7 shows the variation of $R_p$ and $C_{dl}$ for the respective carbide reinforced aluminium tested in this research work. It can be seen (Fig. 8.7 (a and c)) that $R_p$ is found to be higher in Al-2Fe$_3$C followed by Al-2WC, Al-2TiC and lowest for Al-2Mo$_2$C. At the same time $C_{dl}$ is found to be higher in Al-2Mo$_2$C and Al-2TiC and lower in Al-2WC and Al-2Fe$_3$C. Al-2Fe$_3$C and Al-2WC showed greater material resistance to corrosion as Al-2TiC and Al-2Mo$_2$C showed poor material resistance to corrosion. When the carbide concentrations in the aluminium composites were increased from 2% to 4%, the following corrosion behavior is observed. Al-4Mo$_2$C and Al-4TiC showed lower $R_p$ values compared to other composites (Fig. 8.7 (b)). The $C_{dl}$ values seen in Fig. 8.7(d) are inversely proportional to the $R_p$ values seen in Fig. 8.7(b). This indicates that the corrosion is higher in Al-4Mo$_2$C composite and lowest in Al-4Fe$_3$C composite. Same can be seen and verified in the optical micrographs given in Figures 8.3 and 8.4. The $R_p$ and $C_{dl}$ values are in good agreement with the micrographs obtained and hence, can be concretely stated that this corrosion analysis technique can be effectively used for powder metallurgy parts. Further, corrosion in metals usually starts at the grain boundaries and corrosion activity is promoted for powder metallurgy material due to the presence of pores. The pores present allow high pitting corrosion or localized corrosion. A decrease corrosion resistance in the composites is believed to be due to the possible microgalvanic coupling between the reinforced particles or pores and Al particles.
Further, from Fig. 8.7 (a), it is noted that the $R_p$ values remain almost constant even though the content of TiC in aluminium matrix is increased from 1% to 4% except for few cases where $R_p$ values are higher indicating low corrosion in these specimens. Al-3TiC specimens showed lower $C_{dl}$ (Fig. 8.7 (f)) and slightly better $R_p$ when compared to Al-1TiC, Al-2TiC and Al-4TiC. The micrographs also indicate the same that Al-3TiC showed better resistance to corrosion when compared to Al-1TiC, Al-2TiC and Al-4TiC.

Figure 8.7: Variation of polarization resistance and double layer capacitance with respective carbide concentration in aluminum composites, (a and c) 2% carbide concentration, (b and d) 4% carbide concentration and (e and f) 1-4% TiC concentrations. All measurements are taken after 2 hours of immersion.
Further, the effect of initial relative density and percentage deformation is discussed here. Two preforms of 0.82 relative density and 0.86 relative density of each composite is taken and deformed to different height strain, low deformed and high deformed. It can be seen from Fig. 8.7 that the higher initial relative density preform and highly deformed specimen showed greater material resistance to corrosion. This is clearly evident in Fig. 8.7 (a-d) for Al-2Fe\textsubscript{3}C, Al-2TiC, Al-4WC and Al-4Fe\textsubscript{3}C. The effect of initial relative density and final height strain is almost negligible in Mo\textsubscript{2}C reinforced aluminium composites, however, their effect is prominent in WC and Fe\textsubscript{3}C reinforced aluminum composites. This also ties clearly with the micrographs present in Figures 8.3 and 8.4. Similar behavior is found when the TiC content is increased from 1 to 3%, however, the same is not true for 4% TiC content.

The presence of reinforcement in the matrix increases cathode to anode ratio in the composite, resulting in the formation of pits during localized corrosion. The $\beta_C$-parameter can be used to study the pitting corrosion. A higher $\beta_C$ values indicates low corrosion and a low $\beta_C$ values indicates higher corrosion. Figure 8.8 gives the variation of $\beta_C$ for different composites tested in this research work with initial relative density and different levels of height strain. It is seen that $\beta_C$ values are lower for Al-4Mo\textsubscript{2}C and Al-2Mo\textsubscript{2}C composite compared to other composites. It can be noted that the pitting corrosion resistance offered by the specimen is higher in Al-4Fe\textsubscript{3}C and Al-2Fe\textsubscript{3}C. Further, Al-3TiC showed lower $\beta_C$ values when compared to other TiC reinforced aluminium composites. It can be noted that apart from Mo\textsubscript{2}C reinforced aluminium composite, the initial relative density and low deformed Al-4WC and Al-4Fe\textsubscript{3}C showed very high pitting corrosion.
Figure 8.8: Variation of $\beta_C$-parameter with respective carbide concentration in aluminum composites, (a) 2% carbide concentration, (b) 4% carbide concentration and (c) 1-4% TiC concentrations. All measurements are taken after 2 hours of immersion.
From Fig. 8.8 it can be noted that the $\beta_C$ parameter is higher for higher initial relative density preforms and higher height strained preforms, except for Al-4TiC composites. Higher initial relative density preforms usually archives higher final density after even secondary deformation and many researchers have presented the same (Sahin 2003; Eslamian, Rak & Ashgriz 2008; Narayanasamy, Senthilkumar & Pandey 2007; Zhang et al. 2009). This means that the number of pores present is less when final density attainment is high. Further, highly deformed specimen has lower number of pores in comparison to lower height strain specimens. Since the corrosion specimens are taken from the center of the specimen, the pores present are usually round and spherical. These pores act as second phase particles and promote pitting corrosion. Hence, less number of pores are present in higher initial relative density and higher height strained specimens is the reason for high $\beta_C$ values of these specimens suggesting lower pitting corrosion. Same can be seen in Figures 8.3 and 8.4.

More specimens of Al-1TiC, Al-2TiC, Al-3TiC, Al-4TiC, Al-2WC, Al-4WC, Al-2Fe$_3$C, Al-4Fe$_3$C, Al-2Mo$_2$C and Al-4Mo$_2$C were prepared and the testing were carried out at intervals of 2, 4, 6 and 24 hours. The corrosion behavior with respect to immersion time is presented here. A plot seen in Fig. 8.9 demonstrates the effect of composition, strain level and initial theoretical density on the corrosion rate over a period of time for TiC reinforced aluminium composite. Similar plot on Fe$_3$C, Mo$_2$C and WC reinforced aluminum metal matrix composite is presented in Fig. 8.10. A low polarization resistance indicates a high corrosion rate and the results are discussed here. Generally, the corrosion activity decreases rapidly during the start of the experiment and later the corrosion rate increases with time. It is noted that for Al-4Fe$_3$C aluminium metal matrix composite, the corrosion rate decreases rapid during the start of the experiment and then continues to decrease at a steady state rate till the end of the experiment. It is also noted that the corrosion rate decreases up to 4 hours of immersion and then it increases as the immersion time increase. The initial decrease in the corrosion rate up to 4 hours of immersion is higher in TiC reinforced aluminium composites in comparison to Fe$_3$C, Mo$_2$C and WC reinforced aluminium composites. $\beta_C$-parameter given in Figures 8.11 and 8.12 indicates how the surface is taking part in the corrosion activity. A $\beta_C$-parameter of 1
indicates uniform corrosion, meaning the corrosion is taking place on the whole surface and at the same intensity, whereas a $\beta_C$-parameter of less than 1 indicates localized and pitting corrosion. It is further noted for Figures 8.11 and 8.12 that the localized and pitting corrosion decreased up to 4 hours of immersion and after 4 hours of immersion the localized and pitting corrosion is seen to increase. Further, generally it is noticed that $\beta_C$-parameter is around $0.5 \pm 0.2$ indicating more localized and pitting corrosion for majority of the specimens tested.

It was noted that a low deformed specimen and a lower initial relative density specimen had higher corrosion rate when compared to a highly deformed specimens. This is indicated by lower polarization resistance values for lower deformed and lower initial relative density specimens. Further, the corrosion rate was almost constant throughout the immersion time for the lower initial relative density preforms with lower strain levels. Also the $\beta_C$-parameter for these composites was generally found to be constant throughout the immersion time. This was true for all the composites. These preforms had poor final density with maximum pores in comparison to higher deformed specimens having higher initial relative density. The sudden decrease in corrosion rate at the initial stage of immersion and sudden increase in corrosion rate afterwards were mainly exposed by the higher initial relative density preforms and highly deformed preforms.
Figure 8.9: Variation of polarization resistance with immersion time for TiC reinforced aluminum composites.
Figure 8.10: Variation of polarization resistance with immersion time for Fe₃C, Mo₂C and WC reinforced aluminum composites.
Figure 8.11: Variation of $\beta_c$-parameter with immersion time for TiC reinforced aluminum composites.
Figure 8.12: Variation of $\beta_c$-parameter with immersion time for Fe$_3$C, Mo$_2$C and WC reinforced aluminum composites.
8.5 Conclusion

The corrosion behaviors of Al-1TiC, Al-2TiC, Al-3TiC, Al-4TiC, Al-2WC, Al-4WC, Al-2Fe3C, Al-4Fe3C, Al-2Mo2C and Al-4Mo2C were studied in this research work. The following conclusions are drawn:

- The micrographs and electrochemical measurement strongly show that the Mo2C reinforced aluminium composites has very poor material resistance to corrosion. Mo2C reinforced aluminium composites further showed strong pitting corrosion irrespective of initial relative density and final height strain. It is noted that 2Fe3C and 4Fe3C reinforced aluminium metal matrix composite showed better corrosion resistance.

- The corrosion behavior is strongly dependent on the surface porosity. As expected, it was seen that higher porosity specimen produced lower corrosion resistance.

- The galvanostatic pulse technique using a typical three cell electrode configuration is utilized in this study to study the corrosion behavior of powder metallurgy materials. It is strongly noted that this technique can be successfully used for such studies.

- It was found that the corrosion rate of the composites increased rapidly with immersion time during the initial stage. Later it was seen that the corrosion rate decreases with increasing immersion time. This implies that the corrosion resistance of the composites in the present study increases as the exposure time is increased. The phenomenon of reduced corrosion rate with respect to the exposure time shows some passivation of the matrix composite.
Chapter 9
Conclusions & Recommendations

9.1 Conclusion

The densification behavior, workability behavior and corrosion behavior of iron carbide, molybdenum carbide, tungsten carbide and titanium carbide reinforced aluminium metal matrix composites were studied in this research work. The respective compositions were selected based on the extensive literature review and industrial applications.

It is clearly noted that not any particular composition analyzed in this research work stood out best in all three categories, that is, best in densification behavior, best in workability behavior and best in corrosion behavior. Hence, depending on the application and the required properties a best material can be selected from the set of materials tested in this research work. For example, corrosion properties may be absolute essential for a particular application, however, may not require having high final density for the same application. Hence, iron carbide reinforced aluminium metal matrix composite can be used here as it has good corrosion resistance properties even though it may not have the highest final density amongst the composite tested in this research work.

The microstructural analysis was conducted at several stages of this research work. Generally the pores at the edge of the specimen were elongated in the direction of deformation, were higher in numbers and bigger in size. On the other hand, pores at the center of the specimen were round in shape. It is noted that the amount of pores and grain structure varies extensively in the composites tested in this research work.

Pure aluminium preforms major drawback was its very small fracture strain compared to all other aluminium metal matrix composites tested in this research work.

TiC reinforced aluminium metal matrix composites produced the highest density followed by Fe3C reinforced aluminium metal matrix composite. However, they had
lower height strain to fracture compared to Mo$_2$C and WC reinforced aluminium metal matrix composite.

TiC reinforced aluminium metal matrix produced the highest stress ratios followed by Fe$_3$C, then Mo$_2$C and lowest was found in WC containing compacts. Varying the carbide concentrations had no effect on the characteristics behavior and trend of the stress ratio parameters analyzed against density and axial strain. However, the lower carbide concentration preforms produced higher stress ratio parameters. Further, TiC reinforced aluminium metal matrix composites produced good formability stress index over other composites.

Oyane’s fracture model was used to plot the workability limit diagram to identify safe forming zone. The constant in the fracture model was obtained through root mean square techniques and a graph between effective strain at fracture and formability factor at fracture was plotted. In another strategy to plot workability diagram, strain hardening parameters were used to highlight the safe working and non-safe working zones. These plots can be effectively used in the forming process and die design.

The corrosion analysis was carried out using the galvanostatic pulse technique. The corrosion properties of the composites were strongly depended on the surface porosity. Mo$_2$C reinforced aluminium metal matrix composites was more vulnerable to corrosion whilst Fe$_3$C reinforced aluminium metal matrix composites showed strong resistance to corrosion. After a sudden increase in corrosion rate, it was generally seen to reduce over increasing immersion time.

9.2 Recommendations & Future Works

1. It is obvious that the microstructural features, distribution of the reinforcement particles, volume fraction of reinforcement particles and consolidation levels would affect the mechanical properties of aluminium metal matrix composites. The author is keen on carrying out mechanical properties analysis, tensile, impact and hardness measurements, and fractography analysis in his future work. This will help the author to understand the influence of alloying element and forming
process on the mechanical properties of the composite materials tested in this research work. The impact and tensile test fractured specimens can be used for fractography analysis. This will provide the author with evidences of the loading history, the defects caused by friction, alloy compositions or processing technology as well as microstructure characteristics. The necessary changes in the processing technology of the structural parts can be made from these analyses.

2. The author is also keen to evaluate the wear behavior on pure aluminium preforms as well as on carbide reinforced aluminium metal matrix composites.

Wear is one of the most significant tests carried out where the end product is exposed to adhesion, abrasion, and surface fatigue. Metal matrix composites are typically used for tribological applications due to the added advantages over conventional based alloy such as good sliding wear resistance, high load carrying capacity, low density, and improved wear resistance in automotive, aircraft brakes and diesel piston engines (Muratoglu & Aksoy 2000). Muratoglu and Aksoy (2000) investigated friction and wear behavior of 2124-SiC, 2124 aluminium alloy and 1050 steel at different temperatures. They found out that the wear resistance of SiC strengthened metal matrix composites are higher than other compositions chosen in their study for all the test temperatures. The hard SiC particulate which has high wear resistance has enhanced the wear resistance of the metal matrix composite. Bedir (2007) presented that the best wear rate was achieved at 15wt.% and this result is in agreement with the results provided in (Muratoglu & Aksoy 2000). Increasing the volume fraction further than 15% only decreases the wear rate by a very small amount showing insensitivity to volume fraction beyond 15%.

Very little or no work is found on the wear behavior of iron carbide, tungsten carbide, molybdenum carbide and titanium carbide reinforced aluminium metal matrix composites. This is a motivating factor to carry out this work in future.
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Appendix A

Metal preparation technique of aluminium metal matrix composites for microstructure viewing.

1. Cut the specimen into half using a fine hack saw blade.
2. Mounting - Specimens will be hot mounted using a mounting press available in the lab – if required.
3. Grinding - Surface layers damaged by cutting will be removed by grinding; using the emery papers starting with #240, 400, 600, 800 and 1200. Note:
   a. Change the direction of grinding at 90° to that of initial angle of grinding.
   b. If machine is used for this apply load = 22N and speed = 240-300 rpm.
4. Rinse and dry the specimen.
5. Polishing – apply 9 microns diamond paste to the VEGA cloth. Wet the surface using water based lubricant.
6. Polish using the machine for 3-5 min at 22N load at 120-150rpm. After 2 min add 9 microns DP suspension poly crystalline diamond slurry.
7. This is now added every 30 sec until completed.
8. Dry the specimen.
9. Apply 3 microns diamond paste to the Sten cloth. Wet the surface using water based lubricant.
10. Polish using the machine for 3 min at 22N load at 120-150rpm. After 2 min add 3 microns DP suspension poly crystalline diamond slurry.
11. This is now added every 30 sec until completed.
12. Apply colloidal silica to CIAR cloth for 90 – 120 sec, at 120-150rpm in both directions.

13. Last 10 sec apply water to the cloth

14. Clean the polished surface using cotton soaked in soap solution, then with water and then with ethanol using cotton.

15. Dry with warm air.

16. Wash the cloth thoroughly with water.

17. Etching – using tongs apply Keller’s reagent soaked in surgical cotton on the polished surface

18. Clean using distilled water and then with ethanol and dry using a blower.

19. Microstructure viewing
Appendix B

Corrosion test procedure of aluminium metal matrix composite – weight loss method.

1. Weigh the specimen.
2. Fully dip the specimen in the conical flask containing 3.5% NaCl or 1 M HCl. Ensure that no part or sides of the specimen is touching the conical flask. A nylon string (fishing line) is used to help suspend the specimen in the solution as it will not react with the solution.
3. The conical flask is then closed to ensure and avoid atmospheric reaction with the solution.
4. Constant volume of the solution is used for all test samples, approx 400 mls each.
5. The specimen is removed and cleaned in water carefully to remove all dust particles and solution contained in the specimen.
6. Rusted parts are also removed using fine emery paper #600 or 800.
7. Specimens are then heated in the furnace at 50°C for 30 minutes.
8. Weigh the specimens again after cooling to room temperature.
9. Clean the flask and fill with new solution.
10. Repeat steps 2-9 again.
11. The specimens are removed after every 24 hours for the test duration of 96 hours to obtain four sets of results. Each time a fresh solution is used.
Appendix C

Corrosion test procedure of aluminium metal matrix composite – Galvano static technique.

1. Machine the specimen into cylindrical bar having circular cross section area of 1 cm\(^2\) and an height of 0.5-1 cm.

2. **Mounting** - Specimens will be hot mounted using a mounting press available in the lab.

3. **Grinding** - Surface layers damaged by cutting will be removed by grinding; using the emery papers starting with #240, 400, 600, 800 and 1200. Note:
   a. Change the direction of grinding at 90\(^\circ\) to that of initial angle of grinding.
   b. If machine is used for this apply load = 22N and speed = 240-300 rpm.

4. Ensure a circular surface of 1 cm\(^2\) is only exposed.

5. Rinse with distilled water and then with absolute ethanol.

6. Drill a 2 mm hole in the thermoplastic mounting just enough for the copper wire to make contact with the specimen from the back.

7. Seal the hole with adhesive to hold the copper wire in place and avoid solution entering the 2 mm hole.

8. Fully dip the specimen in 3.5% NaCl and ensure part of the copper wire is outside the solution for electrical connection.

9. Dip the reference electrode and the counter electrode to the same submergence level as the specimen as per Fig. 8.2.

10. Supply potential of 1.5 volts between the counter electrode and the working electrode.
11. Apply 1 second galvanostatic pulse and record the potential time response data.

12. Analyze the charging curve of the potential time response data for corrosion results.
Appendix D

Photographs of Specimens

Figure D1: Specimens after the sintering process with aspect ratio (AR) of 0.6, 0.4 and 0.2. For each composition a total of 24 specimens were prepared for workability analysis.
Figure D2: Deformed specimens for workability analysis.

Figure D3: (a) Samples taken for microstructure analysis, (b) Specimens prepared for microstructure analysis.