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Deformation and fracture behaviour of particulate reinforced 7075 aluminium alloy

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University of Wollongong

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DEFORMATION AND FRACTURE BEHAVIOUR
OF PARTICULATE REINFORCED 7075
ALUMINIUM ALLOY

A thesis submitted in fulfilment of the requirements for the
award of the degree of

DOCTOR OF PHILOSOPHY

from
The University of Wollongong

by
Ahmad Razaghian Arani, B.E., M.E.

Department of Materials Engineering

April 1995
Declaration

The work presented in this thesis was carried out by the candidate at the Department of Metallurgy and Materials Engineering of the University of Wollongong, Australia, and has not been submitted to any other universities or institutions for a higher degree.

Ahmad Razaghian Arani
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ABSTRACT

The deformation and fracture behaviour of 7075 aluminium alloy reinforced with 15% vol. of SiC particles and 7075 monolithic alloy were studied. The study involved both compression and tensile modes of deformation at high temperature. The effect of temperature and strain rate on hot workability was examined using uniaxial compression test in the temperature range of 250-450°C and strain rates of 0.001-1 s\(^{-1}\). The results showed that the flow curves exhibited strain hardening at low strain followed by steady state stress at high strain as generally shown in Al-alloys at high temperature. The composite was stronger compared to monolithic alloy at lower temperatures, but both possessed similar strength above 400°C. The higher flow stress at lower temperatures seems to be due to higher dislocation density and slow rate of recovery in the matrix.

The Selected Area Diffraction pattern (SADP) showed that the most subboundaries were low angle boundaries, however, some highly misoriented grains were observed at 450°C. The results indicate that dynamic recovery is the dominant restoration process during deformation in the composite and large SiC particles has no significant effect on deformation behaviour at elevated temperature. The composite showed fine substructure with high dislocation density at lower temperatures up to 300°C, but as temperature increased the dislocation density decreased and the subgrain size increased. The reciprocal subgrain size, d\(^{-1}\), showed a linear relationship with log \(z\). Activation energy was found to be 168 kJ mole\(^{-1}\) which is higher than that for the monolithic alloy (151 kJ mole\(^{-1}\)).

The double pass deformation was carried out in compression at 300 and 400°C at a strain rate of 1 s\(^{-1}\). Between two passes the delay time was varied from 6 to 900 sec to study its effect on fractional softening (FS). It was found that fractional softening increased substantially with increasing temperature but marginal increase in FS obtained when delay time between passes increased. The degree of FS observed in 7075Al/SiCp composite and monolithic alloy was found to be lower compared to other aluminium alloys. This can be attributed to the stabilizing effect of dispersions and fine precipitates which are presented in 7075 Al matrix. The insignificant changes in the microstructure during interpass hold times shows that the low FS in these alloys seems to be due to static recovery. It was found that microstructure and substructure generated in the samples either by continuously or interrupted straining in compression were similar at a given temperature.
In the second phase of high temperature deformation of these materials the deformed samples were annealed at high temperature to study the static recrystallization (SRX) after hot working. The results showed an absence of SRX in the samples annealed after deformation at the same temperature, however, a rise in annealing temperature of 100-150°C above that the deformation temperature led to full recrystallization. This can be ascribed to the relatively moderate dynamic recovery and the presence of dispersions which stabilize the substructure. Particle stimulated nucleation (PSN) had a significant effect on the grain size in deformed samples at low temperature, but no PSN was observed in samples strained at high temperatures. The possible cause might be that at high temperature the dislocation can be annihilated by climb process around the particles together with absence of deformation zone for nucleating the recrystallization.

In the tensile mode of straining at high temperature, the tensile tests were carried out to study the ductility and fracture behaviour of the composite. The tests were performed in the temperature range from room temperature to 400°C at strain rate of 0.001 s\(^{-1}\). Two different conditions of heat treatment were used (T6 and annealed). The results showed that the ductility increased as temperature increased up to 350°C and remained nearly constant as temperature increased to 400°C. The composite showed a much lower ductility compared to the monolithic alloy at all temperatures, although both materials exhibited similar strength levels at temperature above 300°C.

The results of fracture behaviour showed that at room temperature the fracture mechanism changed from particle fracture in T6 condition to interface decohesion in annealed condition. In T6 condition, the fracture mechanism changed from particle fracture at room temperature to interparticle voiding at high temperatures. Particle fracture was found predominantly in cluster of particles regions where large particles were present. This can be attributed to the high local stress in these regions and high probability of flaws in large particles. At high temperature the composite revealed a dimpled fracture surface indicating ductile fracture of the matrix. The internal damage associated with particle cluster was observed in regions below fracture surface and the amount of damage increased with increasing temperature. It was found that the cluster of particles regions and large particles regions were prone locations to permanent damage in these materials at both room and high temperatures.
NOTATION

$\varepsilon$  Strain rate ($s^{-1}$)

$\varepsilon$  True strain

$\sigma$  True stress (MPa)

$m$  Strain rate sensitivity

$\alpha, \beta, A$  Constants

$Q$  Activation energy for Deformation

$n$  Stress exponent

$n_i$  Temperature independent constant

$d_c$  Subgrain size for the composite ($\mu m$)

$d_m$  Subgrain size for the monolithic alloy ($\mu m$)

$R$  Gas constant

$\sigma_{0.2}$  0.2% proof stress (MPa)

$T$  Deformation temperature ($^oK$)

$Z$  Temperature compensated strain rate (Zener-Holloman parameter)

$FS$  Fractional softening
Scope and Contents of This Thesis

Discontinuously reinforced metal matrix composites (DRMMCs) based on aluminium alloys have been confirmed to exhibit improved stiffness, strength and creep resistance while retaining low ductility. These materials can be fabricated by conventional metal working techniques, such as hot extrusion and rolling, thus they have great potential to be used in industry to replace the conventional aluminium alloys and steels for especial purposes. Along with these advantages, however, the low ductility and fracture toughness of these materials, due to the presence of brittle reinforcements, limits usage of these materials in many structural applications.

There has been a considerable research work on strength mechanisms of the MMCs at room temperature, but relatively limited studies have been undertaken on high temperature deformation of these materials especially the hot workability and the dynamic restorations accompanying such deformation. Furthermore, evaluating the effects of reinforcement size and volume fraction on the fracture properties of aluminium based MMCs has been extensively considered. However, limited information is available on the effect of heat treatment on fracture behaviour at room temperature as well as tensile properties and failure mechanisms at elevated temperature in aluminium based DRMMCs. In order to design optimum parameters for hot forming of these materials it is thus important to understand the effect of deformation parameters such as temperature and strain rate under conditions of single and multiple deformation. Moreover, an understanding of the effect of microstructure and temperature on fracture behaviour of these materials is necessary in order to develop maximum fracture resistance at room and high temperatures. The study presented in this thesis is concerned with deformation behaviour of an aluminium alloy based MMC in the temperature range of 250 to 450°C and strain rates of 0.001-1 s\(^{-1}\) as
well as tensile properties and fracture behaviour of the composite at room and elevated temperatures.

The materials used in this study are 7075 aluminium alloy and a composite containing 15% volume fraction of SiC particles with an average size of 14 μm in 7075 aluminium alloy. These materials were supplied by Alcan Canada Ltd in the form of hot extruded rectangular bars.

The present work involves the study of high temperature deformation of reinforced and unreinforced 7075 aluminium alloy during single and double pass deformation as well as fracture behaviour at low and high temperatures. The microstructural examinations are carried out by optical, transmission and scanning electron microscopy.

The content of this thesis, 6 chapters, are briefly described below:

Chapter 1 is literature review and contains five sections. The first section describes the historical background of development of MMCs. Strengthening mechanisms in MMCs including continuum models such as Eshelby and Shear lag models as well as matrix strengthening models suggested by Arsenault and Humphreys are outlined in section two. Deformation mechanisms of the metals at elevated temperature are explained in later section. In this section a detail of restoration mechanisms of dynamic recovery and recrystallization with emphasis on flow behaviour and microstructural development is given. The deformation behaviour and the effect of fine and large particles on restoration mechanisms in aluminium and its alloys are also described. Some limited findings from a few studies carried out on hot working of MMCs are presented at the end of third section. Section four describes the mechanical behaviour and microstructural development after multistage deformation in aluminium alloys. The effect of delay time, temperature, strain and strain rate on fractional softening as well as static recrystallization after hot working in
aluminium and its alloy also are presented. The ductility and fracture behaviour in MMCs at low and high temperature are given based on several studies in section five.

The various experimental techniques and procedures used are described in chapter 2. The mechanical testing including compression and tensile testing as well as the sample preparation are outlined. The procedure for preparation of samples for optical and electron microscopy is also presented.

Chapter 3 is concerned with an investigation of deformation behaviour in a range of temperature from 250 to 450°C at strain rates of 0.001-1 s⁻¹. Constant strain rate testes were performed in uniaxial compression testing. The effect of temperature and strain rate on the flow behaviour, strength and microstructure was studied. Transmission electron microscopy (TEM) was used to examine the microstructural development after deformation. Selected Area Diffraction Pattern (SADP) was utilized to examine the microanalysis of hot worked composite samples.

Chapter 4 reports the effect of temperature and delay time on fractional softening and microstructural development of these materials during double pass deformation in compression. In this work, the compression samples were first strained to a true strain of 0.4, in first pass, held for different times and then compressed again to a further strain of 0.5 thus giving total strain of 0.9. The tests were performed at 300 and 400°C at a constant strain rate of 1 s⁻¹. The delay time between the two passes was varied from 6 to 900 seconds. The results are discussed in terms of the effect of temperature and delay time on fractional softening and the role of fine precipitates on stability of substructure is also discussed. This chapter also considers the static recrystallization after hot working in these materials. The hot deformed samples were annealed at the deformation temperature and temperatures up to 500°C for 1 hour. The results are discussed in terms of the
influence of SiC particles, fine precipitates, deformation and annealing temperature as well as strain rate on recrystallized grain structure

The tensile properties and fracture behaviour of the composite are described in chapter 5. The investigation was carried out using tensile testing under constant strain rate conditions. The tests were carried out in a temperature range from room temperature to 400°C at a strain rate of 0.001 s\(^{-1}\). The tensile specimens were tested in two different heat treatments (T6 and annealed conditions). Optical microscopy and scanning electron microscopy were carried out to study the fracture surfaces and longitudinal cross section of the fractured specimens. The effect of temperature and matrix microstructure on tensile properties and fracture behaviour are discussed in this chapter.

Chapter 6 outlines the general conclusions derived from the study described in previous chapters and suggests future work.
CHAPTER 1
Literature Review
1.1 Introduction to Metal Matrix Composites

Composite materials generally consist of a bulk material called matrix and a filler or reinforcement material of some type, such as fibers, whiskers, particulate, or fabric. The composite materials are usually divided into three broad groups identified by their matrix materials: metal, polymer and ceramic. In metal matrix composites (MMCs), the matrix is usually an alloy and the reinforcement is an intermetallic, an oxide, a carbide or a nitride. The reinforcement materials usually carry the major stresses and loads, while the matrix material holds them together, enabling the stresses and loads to be transferred to the reinforcement materials.

Metal matrix composites are classified into three groups including dispersion strengthened, particle reinforced and fiber reinforced composites. Dispersion strengthened composites are characterized by microstructures consisting of fine particles dispersed uniformly in a matrix. The particle size is very small, with a diameter less than 0.1 μm and volume fractions range from 1 to 15%. The fine dispersions impede the motion of matrix dislocations and produce a pronounced strengthening effect. On the other hand, particle reinforced composite is characterized by dispersed particles with a size greater than 1 μm in diameter and a volume fraction between 5 and 40%. In fiber (whisker) reinforced composites, the size of reinforcing phase has a wide range from 0.1 to 250 μm in length to continuous fiber, and the volume fraction can be between a few percent to as high as 70% (Askeland, 1988; Farag, 1989; Taya and Arsenault, 1989). However, depending on the form of the reinforcement, fiber reinforced composites can be divided into two major groups; discontinuous and continuous. Also MMCs generally are classified into two different major categories, i.e. continuous and discontinuous.
It should be noted that metal matrix composite technology began in the late 1950s. The initial emphasis was on development of continuous filament MMCs because they, with high specific strengths and elastic modulus, could be used for specialized aerospace applications (Lee and Mykkanen, 1987; Kamat et al., 1989). However, these composites are not cost effective for most applications because of the high costs of reinforcement materials, fabrication and secondary processing. This has led, in recent years, to the development of the relatively less expensive new low cost fibers (Divecha et al., 1981) for non-aerospace applications and especially particulate reinforced aluminium-matrix composites for potential use in certain aerospace applications where the very high directional properties available in continuous fiber-reinforced MMCs may not be required (Kamat et al., 1981; McDanels, 1985). Although the strengths of discontinuously reinforced (DR) composites are generally inferior to composites with continuous fiber reinforcement, they have several advantages. Firstly conventional metal working techniques can be used to shape these discontinuously reinforced composites (Nieh et al., 1983; 1984). Secondly, lower raw material costs coupled with the use of conventional metal working techniques can result in a lower cost and finally these composites show isotropic properties in contrast to continuous reinforced metal matrix composites which exhibit anisotropic properties material (Park et al., 1990).

During development of MMCs, many metals have been tested as the matrix alloy, but the most important are aluminium, titanium, magnesium and copper alloys. Among these metals, aluminium alloys are the most common matrix alloys used for discontinuous MMCs (Long et al., 1988; Taya and Arsenault, 1989; Tuler and Klimowicz, 1990). The most prominent discontinuous reinforcements used in MMCs are SiC, Al₂O₃, Si₃Ni₄ and B₄C in both whisker and particulate form. Discontinuous silicon carbide reinforced (short fiber or particulate) aluminium alloy composites have received considerable interest due to several attributes. It has been demonstrated by several studies that the addition of
silicon carbide substantially improves the matrix modulus, and results in enhanced matrix strength (Divecha and Fishman, 1980; Hasson et al., 1985). In the following section, the strengthening mechanisms in discontinuous MMCs are reviewed.
1.2 Strengthening Mechanisms in Discontinuous Reinforced Composites

There is an important distinction between the strengthening mechanisms in continuous fiber MMCs and discontinuous MMCs. In the continuous fiber MMCs, the long fibers carry essentially all of the load and the matrix serves to transmit the load to the fiber (Dieter, 1976; Taya and Arsenault, 1989). In this case, when a load is applied parallel to continuous, unidirectional fibers, the Young's modulus or stiffness for the composite can be determined from the simple "rule of mixtures" addition of the elastic modulus of the matrix and the fiber as:

\[
E_c = E_f V_f + E_m V_m \quad (1-1)
\]

and the yield or flow stress can be expressed as

\[
\sigma_c = \sigma_f V_f + \sigma_m V_m \quad (1-2)
\]

Where \(E_c\), \(\sigma_c\) are the stiffness and flow stress of the composite, \(V_f\), \(E_f\) and \(\sigma_f\) are the volume fraction, stiffness, flow stress of the fiber, \(V_m\), \(E_m\) and \(\sigma_m\) are the values of the matrix.

In continuous fiber composites, the elastic moduli are often anisotropic due to the geometric and material properties of reinforcing phase. Because of the anisotropy of the continuous fiber composite, the \(E_c\) is predicting \(E_L\) (longitudinal modulus of continuous fiber). The law of mixture prediction is also reasonably accurate for aligned short fiber reinforcement but it gives poor estimate of \(E_L\) of misorientated short reinforced MMCs and \(E_T\) (transverse modulus) of continuous and short fiber MMCs (Mura, 1987). In discontinuous MMCs (DMMCs) the elastic modulus depends on ratio of fiber length to fiber diameter or fiber aspect ratio \(\left(\frac{l}{d}\right)\), fiber misorientation and other constituent
properties in addition to volume fraction of reinforcement phase (Jones, 1975; Nardone and Prewo, 1986). In the case of yield or flow stress, although the value calculated by this law is an upper boundary value, it can predict reasonably well the longitudinal properties of unidirectional continuous fiber composites. But in the case of short fiber composites, the law of mixtures value is well above that of the composite.

In summary, the law of mixtures is not suitable to predict the strength of discontinuous fiber or particulate metal matrix composites. This is because, in DMMCs, the ends of each short fiber carry less of the load than the remainder of the fiber. Therefore other models should be used to predict the stiffness of a metal matrix composite. Several models on the basis of a continuum approach, such as shear lag model, Eshelby model and modified shear lag and Eshelby models have been developed to predict the mechanical properties of DMMCs. Micromechanical models based on strengthening the matrix by fibers and particles have been proposed by Arsenault and Humphreys. Arsenault and other researchers (Arsenault, 1984; 1993) have suggested that higher dislocation density due to the presence of fibers or particles is the main reason for the strengthening of DMMCs. In the Humphreys' model, the strengthening mechanism of the DMMCs is considered to be similar to that of conventional two-phase alloys (Humphreys et al., 1991). These strengthening mechanisms will be explained greater detail in the following section.

1.2.1 Continuum Model

In the continuum model for predicting of mechanical properties of short fiber composites, two types of analytical models seem to have been used extensively, the shear lag type and the Eshelby type models.
(a) Shear Lag Model

The shear lag model which was originally developed by Cox (1952) is simple and has been used for prediction of stiffness, yield stress, strength and creep strain rate (Kelly, 1973; Nardone and Prewo, 1986). The standard shear lag model has often been used for an aligned short fiber composite system with short fibers of uniform length and diameter (constant aspect ratio, $\frac{l}{d}$). It is assumed that fibers are distributed in the matrix in hexagonal arrays with all fibers aligned in the loading direction. The most important assumption in the shear lag type model is that load transfer occurs between a short whisker and matrix by means of shear stresses at the matrix-whisker interface. In the original shear lag model, the load transfer by normal stress at the fiber ends and side surfaces was ignored. According to Cox (1952), if the whole of matrix is subjected to a strain, $e$, in the direction of the fiber, the load in the fiber will be:

$$P = E_f A_f e \left[ 1 - \frac{\cosh \beta (l/2 - x)}{\cosh \beta (l/2)} \right]$$  \hspace{1cm} (1-3)

where $E_f$ is the modulus of fiber, $A_f$ is the area of section of the fiber and $h$ is a constant, $l$ is the length of the fiber, $\beta = (h/E_fA_f)^{1/2}$. Assuming that there is no load at the ends of the fiber, $P = 0$; at $x = 0$ and $x = l$; the distribution of tensile stress in the fiber can be obtained as (Cox, 1952; Kelly, 1973):

$$\sigma = E e \left[ 1 - \frac{\cosh \beta (l/2 - x)}{\cosh \beta (l/2)} \right]$$  \hspace{1cm} (1-4)

According to this model (Taya and Arsenault, 1987) Young's modulus of the composite will be:

$$\frac{E_c}{E_m} = (1 - V_w) + V_w \left( \frac{E_w}{E_m} \right) \left( 1 - \frac{\tanh \frac{x}{\lambda}}{x} \right)$$  \hspace{1cm} (1-5)

$$x = \frac{l}{d} \{ (1 + \nu_m) \left( \frac{E_w}{E_m} \right) \ln \left[ V_w \right]^{-1/2} \}^{-1/2}$$  \hspace{1cm} (1-6)
and the yield stress,

$$\sigma_{yc}/\sigma_{ym} = 0.5 V_w (2 + l/d) + (1 - V_w )$$

(1 - 7)

Where $E_m$, $E_w$ and $E_c$ are Young's modulus of the matrix, whisker and composite, respectively, $V_w$ is the volume fraction of whiskers, $\sigma_{ym}$ and $\sigma_{yc}$ are the yield stresses of the matrix and composite, respectively, and $l/d$ is the whisker aspect ratio.

This is the conventional shear lag model to predict the stiffness and yield stress of short fiber composites. Nardone and Prewo (1986) modified the conventional shear lag model by considering the load transfer at the ends of the whiskers to predict yield strength of discontinuous short fiber and particulate MMCs. However, the experimental results show that as the aspect ratio of short fiber decreases, that is the fiber length is shorter with respect to diameter, the approximation made in the modified shear lag model becomes questionable (Taya and Arsenault, 1987; Miller and Humphreys, 1991). Since the geometry of small aspect ratio of short fiber is three-dimensional, this requires a more rigorous mathematical treatment to predict the yield stress of short fiber composites.

(b) Eshelby Model

Eshelby (1957) proposed a short-cut method to solve three-dimensional elasticity problems. The original Eshelby model is based on the assumption that an ellipsoidal inclusion with uniform non-elastic strain is embedded in an infinite elastic body (Eshelby, 1957). When this inclusion with elastic modulus tensor $C_{ijkl}$, is subjected to uniform non-elastic strain, it induces stress fields within and outside the inclusion.
According to the Eshelby procedure, the stress field inside inhomogeneity is given by:

\[ S_{ij} = C_{ijkl}(e_{kl} - e_{kl}^*) \quad (1 - 8) \]

where \( C_{ijkl} \) is elastic modulus tensor of the matrix, \( e_{kl} \) is the total strain and related to displacement and eigenstrain \( e_{kl}^* \) by:

\[ e_{kl} = S_{klmnmn}^* \quad (1 - 9) \]

where \( S_{klmnmn} \) is Eshelby's tensor which is a function of the geometry of the inclusion and the Poisson's ratio when the matrix is isotropic.

Mori and Tanaka (1973) modified the original Eshelby model to account for the interaction between inclusions. Mura (1987) has also summarized the details of the Eshelby model and other models derived from the original Eshelby model. Taya and Arsenault (1987) used the modified Eshelby model to predict the stiffness and yield stress of short fiber composite materials and the following equation was obtained to predict of yield stress:

\[ \sigma_{yc} = c_1 \sigma_{ym} + c_2 e_p \quad (1 - 10) \]

Where \( C_1 \) is a non-dimensional parameter (the yield stress raiser), \( C_2 \) is the work-hardening rate and \( e_p \) is the plastic strain in the matrix along the fiber axis.

Taya and Arsenault (1987) used the shear lag and Eshelby models to calculate the stiffness and yield stress of SiCw/1100 Al composite and compared the results obtained by these models with experimental results. Their comparison shows that the stiffnesses predicted by the shear lag model are always less than those predicted by the Eshelby model and that the shear lag model is a rather crude approximation for smaller \( \frac{l}{d} \); while the Eshelby model gives a better prediction of experimental results. However, in the case of yield stress both models predict a yield stress which is much less than that
experimentally obtained, although the Eshelby model gives good prediction of yield stress for T6* treated SiC/6061 Al composite with experimental results.

In summary, the shear lag model predicts a linear increase of the $\sigma_{yc}/\sigma_{ym}$ with increasing volume fraction, but the experimental data do not agree with the predicted linear relationship (Arsenault, 1988). The Eshelby model is more suitable than the shear lag model to predict the stiffness and yield stress for small aspect ratio of short fiber composites. The experimental data of Arsenault and Fisher (1983) and Yang et al. (1991) indicate that the observed strengthening in composites due to reinforcements is greater than that predicted from continuum models, because in these models the matrix and reinforcement are considered as idealized elastic or elastic-plastic materials and do not take into account microstructural changes in the matrix due to the presence of reinforcement. Hence, the increase in yield stress of the composite must be caused by the increased dislocation density in the composite matrix or other microstructural changes due to presence of reinforcement in the matrix. Therefore it is considered that the continuum model for predicting the strengthening of MMCs provides a crude approximation to the actual behaviour of a metal matrix composite and it is not sufficient to explain the behaviour of composites, so other strengthening mechanisms based on strengthening of matrix caused by the reinforcement should be considered. In the following section, the strengthening mechanisms in the matrix developed by Arsenault and Humphreys will be reviewed.

*T6 treated is solution treated at 540°C for 2 hours followed by water quenching and then aged at 160°C for 18 hours.
1. 2. 2 Strengthening Mechanism in Matrix

(a) Arsenault Model

The strengthening mechanism of short (discontinuous) fiber MMCs has been studied by Arsenault and other workers (Arsenault, 1984; 1991; 1993; Flom and Arsenault, 1985; 1986; Arsenault et al., 1991). It has been found from the above studies, that the yield and flow stresses of DMMCs increase with increasing of both volume fraction and fiber aspect ratio. The yield stress in compression is higher than that in tension due to the residual stress in the matrix and finally yield and flow stresses are strongly dependent upon the fiber orientation with respect to loading direction.

Arsenault has proposed a mechanism of strengthening of composites due to microstructural changes in the matrix. This mechanism is based on the work hardening of the matrix as a result of the difference in the thermal coefficient of expansion (CTE) of the reinforcement and the matrix. The difference in CTE between the matrix and SiC results in the work hardening of the matrix due to an increase in dislocation density and a decrease in subgrain size (Arsenault, 1984). Arsenault and Fisher (1983) proposed that the increased strength observed in Al/SiC composites could be accounted-for by a high dislocation density in the aluminium matrix. This high dislocation density is considered to be as a result of large difference (10: 1) between the aluminium and SiC. Therefore, when the composite is cooled from the elevated temperatures of annealing or processing, misfit strains occur due to differential in CTE at the Al/SiC interface which are sufficient to generate dislocations.

An in-situ high voltage electron microscopy (HVEM) study carried out by Vogelsang et al. (1986) has also demonstrated that a very high dislocation density could be produced in the aluminium matrix upon cooling from 773 to 300 K. However, dislocation
generation due to factors other than CTE differences is also possible, for instance, generation of dislocations during plastic deformation processes of manufacturing, such as extrusion. These dislocations may not be annihilated during annealing and they could be trapped by SiC, resulting in a high dislocation density after annealing (Vogelsang et al., 1986). Although the high dislocation density and the decrease in subgrain size are major factors affecting the strength of the matrix, Arsenault (1988; 1993) has pointed out that other factors such as residual stresses, difference in texture between the composite matrix and the matrix without reinforcement, classical composite strengthening (i.e. load transfer) and dispersion strengthening can contribute to the strength of the matrix.

Arsenault and Shi (1986) have developed a simple model based on prismatic punching to predict the dislocation density due to the differential thermal contraction. With consideration the reinforcement particles with dimensions of height $t_1$, width $t_2$ and thickness $t_3$, the misfit strain due to the difference in the CTE between aluminium and SiC is:

$$\varepsilon = \Delta CTE \cdot \Delta T$$

(1 - 11)

Where $\Delta T$ is the temperature difference. According to this model, the dislocation density due to punching can be given as:

$$\rho = \frac{BA\varepsilon}{b(1 - A)} \frac{1}{t}$$

(1 - 12)

Where $B$ is a geometric constant which is theoretically between 4 (for one dimension very small compared with the other two) and 12 (for equiaxed particles), $t$ is the smallest dimension of the particle and $A$ is the volume fraction of the particles.

If the increase of dislocation density is believed to have contributed to strengthening, the following equation can be used:

$$\Delta \sigma = \alpha \mu b \rho^{1/2}$$

(1 - 13)
When equation \((1 - 12)\) is substituted into equation \((1 - 13)\), the following formula is obtained:

\[
\Delta \sigma = \alpha \mu b \left( \frac{A}{1 - A} \frac{B \varepsilon}{b} \right)^{1/2} \left( \frac{1}{t} \right)^{1/2}
\]  
(1 - 14)

where \(\Delta \sigma\) is the increase in tensile strength, \(\mu\) is the shear modulus of the matrix, \(\alpha\) is a geometric constant, \(b\) is the Burgers vector and \(\varepsilon\) is the misfit strain due to the difference in the CTE between aluminium and SiC. When the morphology of the particles is considered, the following equation is used (Taya and Arsenault, 1989):

\[
\Delta \sigma = 2 \alpha \mu \left( \frac{b A \varepsilon}{1 - A} \right)^{1/2} \left( R \right)^{1/3} \left( 1 + \frac{2}{R} \right)^{1/2} \left( \frac{1}{V} \right)^{1/6}
\]  
(1 - 15)

Where \(V\) is the volume of the particle. When \(R < 1\) it represents whisker morphology, when \(R > 1\) it is a representation of platelet morphology and when \(R = 1\) it is a equiaxed particle which is an approximation of a spherical particle.

It was found that the experimental data agreed well with theoretical prediction from prismatic punching when the particle was small, but this model predicted a lower strengthening for large particle size (Arsenault and Shi, 1986; Arsenault, 1988). It is known that prismatic punching is associated mainly with dislocation generation at the edge length of particle (Taya and Arsenault, 1989). Therefore, discrepancy between the theoretical results and experimental data for large particles could be attributed to the smaller edge length over surface area ratio, meaning that more of the dislocations are generated from the interface area. While medium particles have a large edge length and in this situation more dislocations are generated from the edge of particle size than from the interface.
(b) Humphreys Model

The strengthening mechanism of MMCs has been studied by Humphreys (1988), Miller and Humphreys (1990; 1991a; 1991b). A micromechanism model was proposed which was originally developed for conventional particle containing alloys to predict the strength of MMCs. According to this approach, the strengthening due to the presence of particles depends on various mechanisms: 1) Quench strengthening 2) Orowan strengthening 3) Grain strengthening 4) subgrain strengthening and 5) work hardening.

Because of the large difference in thermal expansion between aluminium and SiC, dislocations are generated on quenching from the recrystallization or solution treatment temperature (Miller and Humphreys, 1991a). The dislocation density (ρ) generated is a function of reinforcement size (d), volume fraction (Fv), thermal mismatch (Δc) and temperature change (ΔT). The strength (σd) may be estimated (Humphreys, 1988) by an expression such as:

\[ \sigma_d = \alpha G b \rho^{1/2} \]  \hspace{1cm} (1 - 16)

Where

\[ \rho = 12 \Delta T \Delta c F_v/bd \]  \hspace{1cm} (1 - 17)

where G is the shear modulus of the matrix, b is the Burgers vector and \( \alpha \) is a constant between 0.5 and 1.

The strengthening of DMMCs by Orowan strengthening by passing of particles by dislocations can be obtained from the following formula:

\[ \sigma_0 = 2 G b / L \]  \hspace{1cm} (1 - 18)

where L is the interparticle spacing. But it has been found that this term has a negligible contribution to strengthening of composite unless the particles are smaller than 1 µm. For
example, it was found that the predicted yield stress increment in Al/SiC composite (d = 3 μm, Fv = 0.17) by particle strengthening was only 6 MPa, compared with the observed yield stress increment of 60 MPa (Humphreys, 1988).

It is known that DMMCs may recrystallize during thermomechanical processing resulting in fine grain size in composites. The recrystallization by particle-stimulated nucleation in materials with particles size larger than 1 μm has been established. The recrystallized grain size with assumption of the nucleation of one grain per particle can be obtained from following equation:

\[ D = d[(1 - F_v)/F_v]^{1/3} \]  \hspace{1cm} (1 - 19)

Since the resulting grain size (1 to 10 microns) is usually considerably finer than that in the unreinforced alloy, therefore it may contribute to the strength of the MMCs according to the Hall-Petch equation:

\[ \sigma_G = K_y D^{-1/2} \]  \hspace{1cm} (1 - 20)

Where \( K_y \) is typically 0.1 MNm\(^{-3/2}\).

Humphreys (1977) has shown that for alloys containing particles, when the ratio of volume fraction to particle size (Fv/d) is more than 0.1 μm, the deformed material will not recrystallize on annealing. In this case, the annealed material will retain a fine substructure which will contribute to the strength of the material. Substructure strengthening will contribute to the strength of the MMC via the Hall-Petch equation with \( K_y = 0.05 \) MNm\(^{-3/2}\). Miller and Humphreys (1990) have suggested that substructure strengthening factor is relatively constant over a range of volume fraction of particle.

It is considered that the work hardening of MMCs is not only increased by dislocation structure formed by quenching, but also by load transfer between the matrix and the
particle by means of Orowan loops at low strains (early stage of deformation) and by creation of geometrically necessary dislocations at higher strains (Ashby, 1966; Miller and Humphreys, 1991a).

Miller and Humphreys (1990) compared the experimentally observed yield stress of a 1050Al/SiCp composite produced by powder metallurgy with different volume fraction (5 to 30%) and particle size (3 to 40 µm) with that predicted from theoretical calculations. Several observations were made from their studies. It was shown that strength increased with increasing volume fraction and for a given volume fraction, fine particles were more effective than coarse particles. A linear effect of quenching temperature on strength was observed, it meant that yield stress increased with increasing quench temperature. The difference in strength between furnace-cooled and water quenched samples indicated the effect of quenching temperature (quench strengthening) on strength of composite. The results were relatively in agreement with the theoretical values, but the measured values were lower than those calculated. A comparison of experimentally measured 0.2% yield stress with values calculated from theoretical considerations consisting of all of the above factors, showed that the Humphrey's model predicted an overestimated value for fine particles and an underestimated value for large particles (Miller and Humphreys, 1991a).

In summary, the comparison of the strengthening of DMMCs based on the models proposed by Arsenault and Humphreys indicates that both models consider the strengthening of matrix due to presence of whiskers or particles. This parameter is not considered in continuum mechanisms. However, they consider different aspects to explain the strengthening of DMMCs, Arsenault has proposed that the strengthening of composite is due to increased dislocation density and decreased subgrain size in the matrix caused by the difference between CTE of aluminium and SiC, while Humphrey's model is based on strengthening of conventional two phase alloys and it considers several factors such as subgrain strengthening and work hardening. Although both models are
suitable to predict the strengthening of MMCs, however, they are still far from ideal. This is because both models consider a uniform distribution of particles with same size, but actually in MMCs, there is a wide distribution in particles size and the particles have very irregular shapes which could possible influence the precipitation process. These parameters are not considered in these models but are of importance.
1.3 Deformation Mechanisms at High Temperature

Discontinuously reinforced metal matrix composites are now recognized as important structural materials, having particularly high modulus, high strength and superior wear resistance. These materials are currently being fabricated by a modified ingot metallurgy process such as stir casting, spray casting and powder metallurgy followed by conventional metalworking processes. One of the prime advantages of the particulate MMCs is that the billets of the composites can be mechanically processed to final shape by using conventional secondary processes such as hot extrusion and rolling developed for the monolithic alloys (Harrigan Jr. et al., 1983; Llyod, 1989a; 1989b; McQueen and Sakaris, 1991). From this viewpoint, the deformation behaviour of the MMCs can be studied by conventional theories of hot working such as analysis of flow stress curves and restoration mechanisms. In the following section, hot working theories in metals such as restoration processes and structural changes which occur during high temperature deformation are reviewed.

1.3.1 Restoration Mechanisms at High Temperature

Static recovery and static recrystallization have been considered as mechanisms of restoration occurring in a cold-worked metal during annealing at high temperature. Annealing is usually carried out in the absence of stress or strain, in which case the recovery or recrystallization that occurs is termed static. However, these restoration processes can also take place under stress and concurrent straining, i.e. under conditions of deformation at high temperature. These restoration mechanisms which take place in the presence of stress are termed dynamic recovery and dynamic recrystallization (McQueen and Jonas, 1975).
Two main softening processes, i.e. dynamic recovery and dynamic recrystallization, have been identified in metals during deformation at elevated temperature. Dynamic recovery occurs in metals with high stacking fault energy, such as aluminium, in which case the stress-strain curve shows a gradual rise to a steady state (Dadson and Doherty, 1991). Dynamic recrystallization takes place in metals with low stacking fault energy, such as stainless steel, copper and nickel. The stress-strain curve goes through a peak and then settles into a steady state. Fig. 1.1 shows the typical flow curves of several metals under conditions of high temperature deformation. It can be seen that low-recovery, dynamic recrystallization metals (top four) show an initial high peak followed by softening to a steady state. However, at similar homologous temperatures, the high recovery metals (lower two) show a steady state stress after initial strain hardening without any peak in flow curve. The two softening processes will be reviewed separately in the following.

1.3.1.1 Dynamic Recovery

The predominant softening mechanism in hot working is dynamic recovery. Dynamic recovery involves movement of dislocations out of their slip planes (Stuwe, 1968). This process is the main softening mechanism in metals with high stacking fault energy, e.g. aluminium, \( \alpha \)-Fe, and most bcc metals where climb and cross slip of the dislocations are relatively easy (McQueen, 1977; McQueen and Jonas, 1984; Sellars, 1992). During hot working, both screw and edge dislocations can leave slip planes to meet dislocations of opposite sign and annihilate. These processes are strongly accelerated not only by high temperature but also by the stresses and the high vacancy concentration which are present in the metal during plastic deformation (Stuwe, 1968). As a result, the misorientation of the subboundaries is maintained sufficiently low so that they never become capable of migrating as grain boundaries of recrystallization nuclei.
Figure 1.1  Typical flow curves for several metals deformed at high temperature

(McQueen and Jonas, 1984)
The schematic true-stress true strain curve for a metal which shows dynamic recovery during deformation at elevated temperature is shown in Fig. 1.2. It can be seen that flow stress rises rapidly and reaches its maximum at low strain and then becomes constant in a regime of steady state deformation (McQueen, 1977). In the strain hardening regime, the dislocation density increases and become entangled and rearrange to form cellular structure. Then the rate of dislocation accumulation diminishes with strain leading to a steady state regime in which the rates of generation and annihilation of dislocations become equal (McQueen and Jonas, 1975; 1984). The steady state stress value depends on both temperature and strain rate. The stress dependence on strain rate at constant strain and temperature can be given by empirical relation as:

$$\sigma(\varepsilon, T) = K \dot{\varepsilon}^m$$  \hspace{1cm} (1 - 21)

where \(\sigma\) is flow stress, \(\dot{\varepsilon}\) is strain rate, \(m\) is the strain rate sensitivity and \(K\) is a constant which depends on the material concerned. Strain rate sensitivity can be obtained from the slope of \(\ln \sigma\) vs \(\ln \dot{\varepsilon}\). It is known that \(m\) increases with temperature, and it is a good indicator of changes in deformation behaviour of the material at different strain rate.

The temperature dependence of flow stress at constant strain rate and strain can be expressed by:

$$\sigma(\varepsilon, T) = C e^{Q/RT}$$  \hspace{1cm} (1 - 22)

A plot of \(\ln \sigma\) vs \(1/T\) will give a straight line with a slope of \(Q/R\) (Dieter, 1976).

The strain rate and temperature dependence of flow stress can be written by a power law relationship at low stresses of the usual form:

$$\varepsilon = A \sigma^n e^{Q/RT}$$  \hspace{1cm} (1 - 23)
Figure 1.2 Schematic flow curve for a metal that shows dynamic recovery during hot working
In the high stress regime, an exponential relationship can be written:

\[ \varepsilon = B \exp (\beta \sigma) e^{-\frac{Q}{RT}} \]  \hspace{1cm} (1-24)

However, a sinh law which is fitted data well in all conditions has been suggested (Hardwich and Tegart, 1961-62; Jonas et al., 1968; Dieter, 1976);

\[ \varepsilon = A \sinh (\alpha \sigma)^n e^{-\frac{Q}{RT}} \]  \hspace{1cm} (1-25)

Where \( \varepsilon \) is strain rate, \( A, \beta \) and \( n \) are constants, \( \sigma \) is steady state stress, \( Q \) is activation energy, \( R \) is the gas constant. If the plot of \( \ln \sinh (\alpha \sigma) \) vs \( \ln \varepsilon \) gives a series parallel lines, it means the data can follow the equation of (1-25). The stress exponent, \( n \), is the slope of this plot. The activation energy for deformation can be obtained from the slope of \( \ln \sinh (\alpha \sigma) \) vs \( 1/T \) which is equal to \( Qn/R \).

The correlation of temperature and strain rate on flow stress (peak stress or steady state stress) can also be represented by the following relation:

\[ Z = \varepsilon \exp \left(\frac{Q}{RT}\right) = A \sinh (\alpha \sigma)^n \]  \hspace{1cm} (1-26)

Where \( Z \) is the Zener-Holloman parameter or temperature compensated strain rate (Farag and Sellers, 1973; McQueen and Jonas, 1990).

1.3.1.2 Microstructural Development During Dynamic Recovery

It is now known that in metals with high SFE (e.g., aluminium) subgrains form in the steady state regime and remain equiaxed and constant in size, misorientation and dislocation density due to the action of work hardening and recovery processes (McQueen and Jonas, 1990; Evangelista et al., 1992; Sellars, 1992). The microstructure of a dynamically recovered metal consists of equiaxed subgrains in elongated original grains.
(McQueen et al., 1991a). A well-defined substructure developed in aluminium is shown in Fig. 1.3.

It has been established that the subgrain size increases as the deformation temperature increases or strain rate decreases (McQueen and Jonas, 1975). At a constant strain rate, the observed increase of subgrain size with temperature is due to increased thermal activation which makes the dislocations more mobile and hence capable of responding to the stress fields of more distant dislocation networks and of travelling greater distances to combine or to be annihilated. It is considered that the effect of decreasing of strain rate is to permit a greater number of thermally activated events to occur (Jonas et al., 1968).

The dependence of mean subgrain diameter, $d$, developed during dynamic recovery, on temperature, $T$, and strain rate, $\dot{\varepsilon}$, can be expressed by the relationship:

$$d^{-1} = a + b \log Z$$  \hspace{1cm} (1-27)

Where $a$ and $b$ are empirical constants and $Z$ is Zener-Holloman parameter.

The steady state flow stress, $\sigma$, is related to the subgrain size by the following relation:

$$\sigma = c + e d^{-1}$$  \hspace{1cm} (1-28)

where $c$ and $e$ are constants and $d$ is the subgrain diameter (McQueen, 1977; McQueen and Jonas, 1984).

The mechanisms of dynamic recovery at elevated temperatures usually include thermally activated processes such as climb, cross slip and unpinning of attractive junctions (McQueen and Jonas, 1975). Therefore any factors which affect dislocation movement may affect the level of recovery. Besides temperature and strain rate, some important factors which influence the recovered substructure are stacking fault energy, solute atoms and second phase particles (Cotterill and Mould, 1976). The stacking fault energy has an important effect on the level of recovery, because this controls the ability of dislocations
Figure 1.3  substructures produced in commercial purity aluminum by extrusion at a mean strain rate of $1 \text{s}^{-1}$ a) $250^\circ\text{C}$ b) $350^\circ\text{C}$ c) $450^\circ\text{C}$ (McQueen and Jonas, 1975)
to climb or cross slip (McQueen et al., 1991a). In metals with low SFE such as Cu and γ-Fe, the dislocation mobility is impeded and the balance between dislocation generation and annihilation changes. The dislocation density continues to rise, and results in limited dynamic recovery in these metals (McQueen and Jonas, 1984; McQueen et al., 1991a). This eventually creates nuclei and provides driving force for dynamic recrystallization (Jonas et al., 1969; McQueen and Jonas, 1975).

Solute atoms may reduce recovery either by reducing the SFE or by providing atmospheres which reduce dislocation mobility and possibility diminish climb by binding vacancies (Sheppard et al., 1983; McQueen and Bourell, 1987). Several investigations have shown decrease of stacking fault energy with the addition of solute atoms (Gay and Kelly, 1953; Howie and Swann, 1961).

Particles reduce recovery by pinning dislocations in the walls and by locking the subgrain boundary nodes (Evangelista et al., 1983; McQueen et al., 1984; 1986; Usui et al., 1986a; 1986b). The effect of particles on dynamic restoration mechanisms will be discussed in detail later.

1. 3. 2 Dynamic Recrystallization

During hot working, all metals undergo dynamic recovery, but when climb and cross slip are not easy processes another mechanism intrudes at larger strains. In the case of austenite and other fcc metals with low SFE (in which the rate of softening due to recovery is slow in comparison with aluminium) the work hardening can not be balanced only by dynamic recovery (McQueen, 1982). The dislocation density is gradually increased, and finally recrystallization starts to occur during deformation. When the misorientation between subgrains reaches approximately 10°, the subgrains act as recrystallized nuclei and dynamic recrystallization operates as the softening mechanism.
The microstructure consists of recrystallized grains with substructure (McQueen and Jonas, 1975; McQueen et al., 1991a).

The schematic true-stress true-strain curve is shown in Fig. 1.4. It can be seen that the stress-strain curve goes through a peak and settles into a steady state at high strain. The true stress is lowered beyond the peak stress by dynamic recrystallization and reaches a certain steady-state stress level at which the work hardening and softening (due to recrystallization occurring repeatedly) are balanced (Sellars, 1992). The range of strain up to $\varepsilon_p$ forms the work hardening region, while the region between $\varepsilon_p$ and $\varepsilon_s$ is called partially dynamic recrystallization region, and the strain range after $\varepsilon_s$ is the range of steady dynamic recrystallization (Tamura et al., 1988). The strain hardening stage terminates at the stress peak due to the appearance of sufficient dislocation-free grains which leads finally to flow softening. The dynamic recrystallization starts at a strain around $0.7\varepsilon_p$ ($\varepsilon_p$ is the strain showing the peak stress).

The rate of deformation can influence the occurrence of dynamic recrystallization and change the flow stress curve shape. At high strain rates, the flow stress rises to a maximum at the peak strain. Then, as a result of dynamic recrystallization, it diminishes to a value intermediate between the yield stress and the maximum or peak stress. While at low strain rates, the softening produced by dynamic recrystallization is followed by renewed hardening and a cyclic flow curve due to recurrent cycles of recrystallization occurs as shown in Fig. 1.5 (McQueen and Jonas, 1975). The peak strain in the case of high strain rate is greater than that in the case of low strain rate. When the strain rate is low, the recrystallization proceeds to completion. Once recrystallization is complete, the dislocation density builds up again, raising the flow stress until recrystallization is again nucleated. But in the case of high strain rate deformation, before recrystallization is complete, the dislocation densities at the centers of the recrystallized grains have increased.
Figure 1.4  Schematic flow curve for a metal that shows dynamic recrystallization

(Tamura et al., 1988)

Figure 1.5  Continuous and repeated flow curves showing dynamic recrystallization
during hot deformation (McQueen and Jonas, 1975)
sufficiently so that another cycle of nucleation occurs and new grains begin to grow again. Thus, at any instance, there is a distribution of regions with different degrees of deformation. This results in maintaining the average flow stress at a steady state value.

It is known that the peak stress ($\sigma_p$) and the steady state stress ($\sigma_s$) change with the Zener-Holloman parameter, $Z$, in metals which display dynamic recrystallization. $\sigma_p$, $\varepsilon_p$, $\sigma_s$ and $\varepsilon_s$ are increased with increase in $Z$ (i.e. with increase in $\dot{\varepsilon}$ or decrease in $T$). This follows a similar trend to metals which undergo dynamic recovery. Here, the difference is that $\sigma_p$ and $\sigma_s$ are equal in dynamic recovery due to steady state value in the flow curve.

1.3.3 Dynamic Restoration Mechanisms in Aluminium and Its Alloys

There is a large body of literature on deformation behaviour of aluminium and its alloys at elevated temperature (e.g., McQueen, 1977; 1980; 1981; 1991a; 1991b; McQueen and Conrod, 1986; McQueen and Evangelista, 1988; Kassner et al., 1989). It has been demonstrated that aluminium and its dilute alloys, exhibit the highest levels of dynamic recovery. This gives rise to a polygonized substructure and no evidence of dynamic recrystallization in aluminium and its alloys up to 4% Mg. The absence of dynamic recrystallization in aluminium and its alloys is due to the high level of dynamic recovery which maintains the dislocation density at a level so low that nuclei can not form (McQueen and Mecking, 1984; McQueen, 1987; 1991a). Even when aluminium contains large constituent particles, the finer dislocation cells generated around the particles do not lead to an increase in density because there is sufficient recovery.
During steady state the microstructure exhibits elongated grains which contain substructure. The mean subgrain size, $d_s$, depends on $\log Z$ through equation (1 - 27), and the flow stress on, $d_s$, according to equation (1 - 28). As $Z$ decreases (temperature increases or strain rate decreases), subgrain size increases.

The basic characteristic of dynamic recovery in aluminium persists at strains of up to 60 (McQueen et al., 1985; 1989; Solberg et al., 1989). For example, McQueen et al (1985) found that the subgrain size and misorientation remained substantially constant over strains from 1 to 60 in Al deformed at 400°C. As strain increases the grains become elongated and average grain thickness decreases. When the grain thickness falls below the subgrain size, the grains develop serrations and become pinched off (McQueen et al., 1985; 1991a). The structure thus consists of several subgrains surrounded largely by high-angle grain boundaries (Kassner, 1987; 1989). This type of structure has been misinterpreted as that which occurs during dynamic recrystallization. However, it is not truly recrystallization since the grain boundaries have not migrated more than, $d$, as for discontinuous; nor have subgrain boundaries increased substantially in misorientation as for continuous. As a result, this mechanism has been called geometric dynamic recrystallization. The maintenance of an equiaxed stable substructure over the strain from 0.7 to 60 can be explained by the theory of repolygonization. According to this theory, the subboundaries disintegrate and reform at the equilibrium spacing by the removal or addition of individual dislocations (McQueen and Jonas, 1975; 1990).

Although, it is considered that dynamic recovery is the only restoration process in Al alloys, dynamic recrystallization has been observed in Al with 5 or more than %5 Mg (Sheppard and Tutcher, 1980; McQueen et al., 1984; Lee and McNelly, 1986). Gardner and Grims (1979) found dynamic recrystallization in Al-%5 Mg, Al-%10Mg and Al-%10 Zn when these alloys were compressed at a relatively low strain rate (about $10^{-3}$s$^{-1}$) to a reduction of 50%. The flow curves under these conditions showed a marked peak
and a gradual reduction toward a steady state. It was suggested that the reduction in stacking fault energy by the solute atoms was the reason for the occurrence of dynamic recrystallization in these alloys during hot working. The results of Ueki et al (1986) showed that dynamic recrystallization occurred in Al-5Mg alloy only at strain rates below \(1 \text{s}^{-1}\) at high temperatures. While Sheppard et al (1979) did not observe dynamic recrystallization in Al-5% Mg after extrusion (high strain rates) at elevated temperature.

The apparent conflict in two sets of results appears to arise from the difference in strain rate. At higher strain rates, the solute atmospheres are left behind due to higher dislocation velocities, leading to dislocation densities which are too low to initiate dynamic recrystallization (DRX). But at low strain rates, the dislocation movement is slowed by high solute drag and not by a decrease in SFE. This leads to high dislocation density to start DRX (McQueen, 1981).

Sheppard and Tutcher (1980) found a duplex deformation structure consisting mainly of dynamically recovered subgrains but modified by DRX which occurred primarily at original grain boundaries and at second-phase particles in Al-5Mg-0.8Mn during extrusion. Sheppard et al (1983) also reported that dynamic recrystallization was a dominant deformation mechanism operating in Al-7% Mg during extrusion. It was suggested that the occurrence of DRX was caused by the presence of second-phase particles and possibility high-solute additions, but not by the changing of stacking fault energy. Another microstructural condition which could lead to recrystallization would be a dispersion of precipitates, which raise the dislocation density without inhibiting boundary migration as observed in Al-5Mg-0.8Mn (McQueen et al., 1984).
1.3.3.1 Influence of Particles on Dynamic Restoration Process in Aluminium Alloys

In particle containing alloys subjected to annealing after cold working, the particles can influence the static recrystallization. They can either stabilize the substructure, or not, and hence either retard or enhance recrystallization (Evangelista et al., 1983; McQueen and Jonas, 1984). The influence of particles during hot working is of considerable scientific interest because the particles can simultaneously influence the stress-driven dislocation displacements, the degree of dynamic recovery and the progress of dynamic recrystallization. They can also influence the hot strength and ductility, and the retention of substructure or grain refinement in the product. Several studies have shown that the effect of particles on dynamic softening during deformation under hot working depends on the particle size, strength, and spacing in a manner similar to their behaviour in cold working and annealing (Sheppard and Tutcher, 1980; 1987). Very fine particles (about 0.01 μm) which are closely spaced may inhibit formation of cellular substructure, while fine particles (0.01-0.3 μm) which are closely-spaced particles can pin the subboundaries and stabilize the substructure (Khalish and Lefebvre, 1975; Moris et al., 1976; McQueen, 1980). However, the presence of large particles (> 0.6 μm) can give rise to regions of high strain gradients containing small, highly-misoriented subgrains which leads to the occurrence of dynamic recrystallization (Sheppard and Tutcher, 1981; McQueen et al., 1984).

(a) Effect of Particles on Dynamic Recovered Structure

Particles reduce recovery by pinning dislocations in the walls and by locking the subgrain boundaries nodes. This is caused by interaction of particles with dislocations and cell
walls during hot working. The effect of particles on dynamic recovery is similar to the influence of particles on static recovery after cold working and depends on particle size and distribution.

It has been demonstrated that a dispersion of fine precipitates generally stabilize the substructure and frequently determines its dimensions (Oblak and Owczarski, 1972; McQueen and Jonas, 1975). For example, the dispersoid aluminium alloys such as Al-0.65Fe and Al-0.5Fe-0.5Co exhibited considerably higher strength than aluminium at 200 and 300°C but not much greater at 400 to 500°C (Conrod and McQueen, 1987; Avramovic-Cingara and McQueen, 1988; McQueen, 1991a). At 200°C, the cells in these alloys were smaller than for aluminium, whereas at 400-500°C the subgrains were comparable in size. Microstructural examinations showed the particles were largely in the cell walls at 200 and 300°C, while at 400 and 500°C more than half the particles were inside the subgrains, usually being with a few dislocations. Humphreys and Kalu (1987) studied the effect of temperature and strain rate on substructure in aluminium containing second phase particles. It was found that at higher strain rates and lower temperatures, the subgrains were smaller near the particles than those in the matrix, while at low strain rates and high temperatures, the size of subgrains were similar both close to, and away from the particles. This is because at lower temperatures, dislocations interact with particles and produce extra dislocations, but at higher temperatures, the particles have no effect on microstructure because the dislocations can remove themselves from around the particles by climb or cross slip. This is considered as a main reason for the observed strength decrease as deformation temperature increases in dispersion strengthened alloys. Even though these results have been found in aluminium alloy containing large particles at high temperature, it is possible that a similar loss of stabilizing effect due to fine particles on substructure will be observed at high temperature.
(b) Effect of Particles on Dynamic Recrystallized Structure

It has been identified in general, that recrystallization is retarded or completely inhibited by closely spaced fine particles (Hansen and Bay, 1981; Castro-Fernandez and Sellars, 1988), while coarse particles which are widely-spaced stimulate nucleation of recrystallization during annealing after cold working (Humphreys, 1979; Chan and Humphreys, 1984). It has been found that particle sizes greater than 1 μm are required for particle stimulated nucleation (Humphreys, 1979; Demarkar and Pelissier, 1983; Zaidi and Sheppard, 1983;).

Dynamic recrystallization has been reported in Al-5Mg-0.8Mn after strains of 5 at above 400°C at strain rate less than 1 s⁻¹ (Evangelista et al., 1983, McQueen et al., 1984). This alloy contains Al₆Mn in the form of constituent particles (> 1 μm) and fine particles precipitated in the solid (< 0.2 μm). Dynamic recrystallization appears to be result from enhancement of nucleation by the large particles (>1 μm) (Sheppard et al., 1979; McQueen and Jonas, 1990). As in static recrystallization, the nuclei grows in the regions of fine highly misoriented cells which have formed around these large undeforming particles (Humphreys, 1982; Evangelista et al., 1983; McQueen, 1991a). These grains initially grow rapidly but are usually stopped at a fairly small size by the fine particles. This indicates that the presence of fine particles can retard dynamic recrystallization by pinning subgrain and grain boundaries, whereas large particles (> 0.6 μm) result in dynamic recrystallization. While in Al-1Mg-1Mn, dynamic recovery was the only softening process during deformation at a temperature range of 270-480°C at strain rate of 5 s⁻¹. It was observed that small particles of about 0.1 μm existed in this alloy and with small dynamically recrystallized grains forming only adjacent to coarse intermetallic
particles during high temperature deformation above 450°C (Castro-Fernandez and Sellars, 1988).

The occurrence of dynamic recrystallization in Al-5Mg-0.8Mn and the absence of dynamic recrystallization in Al-1Mg-1Mn during high temperature deformation can be explained by the level of solute atoms, since both alloys contain of Mn₆Al particles. It is known that the high solute atoms increase pinning efficiency of dislocations, and increase possibility of dynamic recrystallization. DRX has also not been observed in pure aluminium, even when it contains large constituent particles (McQueen, 1991a). This indicates that the presence of large second phase particles in aluminium alloys can lead to dynamic recrystallization, only when the alloy contains of more than 5% Mg. Otherwise, the presence of large particles can not cause dynamic recrystallization and dynamic recovery is the sole deformation process during high deformation.

1. 3. 4 High Temperature Deformation of Aluminium MMCs

1. 3. 4. 1 Mechanical Behaviour of Aluminium Based MMCs

The mechanical behaviour of particulate reinforced aluminium based MMCs at elevated temperatures is of interest for two main reasons. Firstly, these composites can be produced as billets, which are subsequently mechanically processed by conventional metal working processes such as rolling and extrusion. Since the ductility of the composites is limited at low temperatures, the processing of these materials is thus usually carried out at high temperatures. Secondly, some composites are of interest for creep resistant structural applications.

The hot workability of MMCs based on 6061, 2124, 5083 and 7090 aluminium alloys reinforced with whiskers or particles of SiC or Al₂O₃ has been studied by torsion and
compression tests in the temperature range of 200-600°C (e.g., Tuler and Beals, 1988; Chandra and Yu, 1991; Xia et al., 1993a; 1993b). The results from these studies show that the strength of the composites and the monolithic alloys decreases with increasing temperature and decreasing strain rate. However, the decrease in flow stress with temperature is faster in the composites compared to the matrix alloys. The composites are stronger than the monolithic alloy at low temperatures, but the difference in strength between the composite and the matrix alloys decreases as temperature increases so that both the reinforced and unreinforced alloys show similar values at above 500°C. While Chanda et al. (1991) reported that the Al-4Li alloy was slightly stronger than the Al-4Li reinforced with 8% vol. SiC particles at a temperature range of 325-525°C and strain rates of $10^{-5}$ to $10^{-1}$ s$^{-1}$.

The flow curves of the composites obey the normal high temperature behaviour of aluminium alloys (i.e. after the limited strain hardening at very low strain, steady state flow occurs). This is indicative that dynamic recovery takes place in these materials during deformation at elevated temperatures. However, Xiong et al. (1990) found a peak in the true-stress true-strain curve in 6061Al/SiC$_w$ composite but not in the monolithic alloy when the compression direction was parallel to the whisker direction. But, a similar flow curve in the composite and the matrix alloy was observed when the compression direction was normal to the direction of orientation of whiskers. It was suggested that whisker rotation was the cause of the peak, and not dynamic recrystallization. The results of Sakaris and McQueen (1992a; 1992b) on hot workability of 6061Al/SiCp and A356/SiCp composites showed that these materials exhibited a peak in the flow curve followed by a degree of flow softening to a steady state regime before fracture at 300°C, whereas the matrix alloys showed a steady state flow curve without any peak. This was attributed to some dynamic precipitation and coalescence accompanied by larger deformation heating than at higher temperatures.
The activation energy for deformation was calculated by using the power law creep equation (e.g., Pickens et al., 1987; Sarkar and Surappa, 1993) and sinh Law (Chandra and Yu, 1991; Sakaris and McQueen, 1992a; Alunni et al., 1993). In the power law equation, the slope of the plot of ln $\sigma$ vs $1/T$, is equal to $Qm/R$, where $m$ is strain rate sensitivity which can be determined from the slope of ln $\sigma$ vs ln $\dot{\varepsilon}$ at different temperatures, $Q$ is activation energy and $R$ is universal gas constant. Several studies have reported when the law equation was used, the plot of ln $\sigma$ vs $1/T$ was not linear for the composite, while it was linear for the monolithic alloy (Pickens et al., 1987; Tuler et al., 1988). It was suggested that the deformation process was too complex to be characterized by a single activation energy in the composite. The sinh equation is suitable for the whole stress level range and can be used to give a single activation energy for the composite. Results indicate that the activation energy of the composites is higher than for the monolithic alloys, independent of whether the law or sinh equations are used to estimate the activation energy. The higher activation energy in the composites is considered to be due to a higher strength of composites at lower temperatures compared to the matrices alloy, and similar flow stress in reinforced and unreinforced alloys at higher temperatures.

3.4.2 Microstructural Development in Aluminium Based MMCs

Several studies have shown that when the aluminium based MMCs are deformed at elevated temperature, the matrix strain hardens slightly. This is primarily due to restoration by dynamic recovery which results in the formation of large subgrains with low energy dislocation arrays (e.g., Dutta et al., 1990; Sakaris and McQueen, 1992b). This occurs because during deformation at high temperature, dislocations can climb around the particles and thus accumulation of dislocations will not occur. In this
situation, no deformation zones will form and the dislocation structure in the composite will be similar to that of the unreinforced alloy. The comparison of substructures of the composite and the matrix alloy shows that the average level of dynamic recovery is lower in the composite compared to the matrix alloy and the reinforced alloy has finer substructure than that the matrix alloy. Although the dominant deformation mechanism in Al/SiC composites seems to be dynamic recovery at elevated temperature, dynamic recrystallization has also been observed in some aluminium MMCs under certain conditions. For example, Tuler et al. (1988) found fully recrystallized structure in 20% vol. SiCp/6061Al composite deformed at a strain rate of 5 s⁻¹ at 500°C, but this did not occur in 10% vol. SiCp/6061Al composite under identical conditions. Xia et al. (1993a) also found some highly misoriented grains (> 25°) around the SiC particles in 15% vol. SiCp/6061Al composite at 500°C and 1 s⁻¹. It should be noted that dynamic recovery is still considered to be the principal deformation mechanism in this material at high temperatures.

In summary, the results from various researches indicate that the dynamic recovery is the principal deformation mechanism in aluminium alloy composites at elevated temperature. However, dynamic recrystallization can possibly occur at high temperatures and at faster strain rates in the composites which contain a high volume fraction of SiC particles.
1.4 Multistage Deformation at High Temperature

The high ductility and low flow stress experienced by aluminium DRMMCs during hot working indicate that they could be fabricated by hot forming techniques economically compared to cold working processes because the soft matrix will be able to flow around the hard particles without damaging them or developing fissures (McQueen and Sakaris, 1991). In most hot working operations such as rolling and forging, the overall shape is attained through a series of deformation steps. These processes generally include rest intervals between various passes or steps. The metals are therefore subjected to restoration processes such as static recovery and recrystallization during any hold or interpass interval at elevated temperature. Several investigators have studied the multistage deformation behaviour of aluminium and its alloys by using torsion and compression tests at elevated temperatures (e.g., Farag et al., 1968; Cotner and Tegart, 1969). Although several studies have been carried out on the hot workability of aluminium based composites, limited information is available on multistage deformation of these materials. In order to design optimum parameters for hot forming of these DRMMCs it is important to understand how temperature, strain and strain rate will influence the deformation behaviour of these materials under conditions of single and multiple deformation. In the following chapter a review of literature relating to multistage deformation is presented.

1.4.1 Determination of Fractional Softening at High Temperature

The fractional softening observed in metals is generally determined from the drop in flow stress during a holding interval and is reported with the reference to the strain hardening...
that occurred during the prior deformation according to the following formula:

\[ F_{Sn} = \frac{\sigma_{un} - \sigma_{r(n+1)}}{\sigma_{un} - \sigma_{y}} \]  

(1.29)

Where \( F_{Sn} \) is the fractional softening during the \( n^{th} \) interval following the \( n^{th} \) pass, \( \sigma_{un} \) is the stress just before unloading in the \( n^{th} \) pass, \( \sigma_{r(n+1)} \) is the yield stress on reloading in the \((n+1)^{th}\) pass and \( \sigma_{y} \) is the yield stress in the first pass. The schematic representation of a double pass deformation flow curve and parameters used to calculate fractional softening is shown in Fig 1.6. In aluminium and its alloys, it has been found that the flow curves in sequence passes show a lower yield stress on reloading than those on unloading (Evans and Dunstan, 1971; McQueen et al., 1989b). This is attributed to the occurrence of static restoration during holding time between passes. The amount of softening which occurs during the intervals in multi-stage deformation has a significant effect on the mechanical behaviour of the metals. Fractional softening during an interval between passes depends on the history of previous deformation passes, such as the amount of strain and rate of deformation, temperature and duration of delay time. The effect of each these parameters on fractional softening will be dealt with in the following section.

(a) Effect of Temperature

The softening which occurs during delay time between passes, or after deformation at high temperature, is due to the static restoration, such as static recovery and static recrystallization (McQueen and Jonas, 1985). Static restoration mechanisms are thermally activated processes which depend on both temperature and time. As temperature increases in a given time, static restoration increases because of increasing thermal activation, and hence the amount of fractional softening increases. Although the
Figure 1.6 Schematic representation of double deformation and parameters used to determine fractional softening

\[ X = \frac{\sigma_2 - \sigma_3}{\sigma_2 - \sigma_1} \]

Where

- \( X \) is fractional softening
- \( \sigma_1 \) is the yield stress of the first deformation
- \( \sigma_2 \) is the flow stress at the end of the first deformation
- \( \sigma_3 \) is the yield stress of the second deformation
increasing temperature leads to a lower stored energy or driving force for static restoration because of decreasing dislocations density and larger subgrains, the effect of temperature on thermal activation is more important than the decrease in stored energy.

The increase in %FS with temperature has been observed in Al and its alloys during interval between passes in multi-stage deformation. For example, fractional softening increased from ~ 30 to ~45% in Al-0.5Fe and from 20 to 25% in Al-0.5-0.5Co as deformation temperature increased from 400 to 450°C (McQueen et al., 1989b). A similar trend has also been observed in Al and Al-Mg alloys (Evans and Dunstan, 1971; McQueen and Ryum, 1985).

(b) Effect of Delay Time

The duration of delay time between passes has a pronounced effect on the amount of softening. Static restoration processes are diffusion controlled, as time increases more diffusional events occur, producing more static restoration, which in turn increases fractional softening in the material. McQueen et al. (1989b) found that in Al-0.5Fe, fractional softening was about 20% with a delay time of 20 sec, as time increased to 100 sec, at 400°C, fractional softening increased further to 37%. Results from several studies showed a linear increase in FS with logarithmic delay time in Al and its alloys (Evans and Dunstan, 1971; Sellars et al., 1986).

(c) Effect of Strain Rate and Strain

The driving force for static restoration during holding time derives from the stored energy, or dislocation density, attained during deformation within the metal (McQueen and Jonas, 1975). Increasing strain rate leads to reduced time for dynamic restoration during deformation, and a higher dislocation density, or greater driving force. These
conditions result in an increase in the amount of fractional softening. An increase in fractional softening from 25 to 65% has been reported (Evans and Dunstan, 1971) in pure aluminium deformed at 410°C when the strain rate was increased from 2.7 to 45 s⁻¹, with an interpass hold time of 10 sec.

The effect of strain on fractional softening in Al, Al-0.5Mg and Al-1%Mg has been studied by McQueen and Ryum (1985). It was found that as the strain in the first pass was raised from 0.4 to 0.8, the %FS increased from 48 to 95% for pure aluminium and from 45 to 50% for Al-0.5Mg; a delay time of 20 sec was used at 375°C with a strain rate of 0.8 s⁻¹. It should be noted that for a true strain of 0.4 the strain hardening regime was apparent at this temperature, whereas for a strain of 0.8 the steady state regime had been obtained. A similar trend was observed by Sellars et al.(1986) in Al-1%Mg using plane strain compression tests.

1. 4. 2 Microstructural Development During Multistage Deformation in Aluminium and Its Alloys

Several investigators have examined the microstructure and substructure in aluminium and its alloys during and after multiple deformation by optical and transmission electron microscopy (e.g, Farag et al., 1968; Evan and Dunstan, 1971; McQueen et al., 1984). Farag et al (1968) used various deformation schedules on super aluminium such as; interrupted isothermal deformation, delay times from 5 to 120 sec between passes, incremental and continuous temperature changes to simulate hot-working operations. It was found that for interrupted isothermal deformation, uniform subgrains after deformation, at various temperatures, were similar to those observed after continuous deformation, although sub-boundaries were more clearly defined. But when deformation was carried out at higher temperature followed by deformation at a lower temperature, the
microstructure after the second deformation was dependent on the prior deformation temperature. For example, if complete recrystallization occurred during interpass holding, the final microstructure was found to be uniform. But if recrystallization was not complete in the interpass period, evidence of the prior deformation substructure was present in the final microstructure.

Evans and Dunstan (1971) have investigated microstructural changes during delay time between passes in commercial pure aluminium. Experiments were conducted by double-stage deformation in the plane strain compression mode. In their experiments, a wide range of delay times from 1 to 4100 sec were used, at temperatures of 330, 410 and 480°C, and with strain rates of 2.7, 8 and 45 s⁻¹. Samples were given a true strain of 0.37 in the first pass, held for different delay times and then compressed to a total strain of 0.6 at a given deformation temperature. Elongated grains containing banded subgrains were observed in a sample held for short time after the first pass at 410°C deformed at strain rate of 2.7 s⁻¹. Subboundaries were ragged with a considerable number of dislocations present within the sub-cells. With increasing delay time, subgrain walls became less ragged and dislocation density, within the subcells, decreased sharply after 80 sec. Small recrystallized grains with well-defined boundaries within the subgrains arrays were also found after a very short delay time and the amount of recrystallized grain increased with further holding time. It should be noted that the subgrain size and the volume fraction of recrystallized grains are dependent on temperature at which straining was carried out.

The microstructure of 1100Al alloy after different passes in multistage rolling has been examined by Sheppard et al. (1986). In their work, the temperature and strain rate at each pass was varied. Temperature was decreased from 460°C in the first pass to 345°C in the third pass, with the strain rate increasing in each sequence pass. Results showed that the substructure was well-defined after each pass and the subgrain size was dependent on
both temperature and strain rate. It was also found that multistage deformation had no significant effect on substructure, the final pass determining the substructure size. Considerable subgrain growth during the interval between passes was also observed, and the extent of growth was dependent on the subgrain size from the previous pass and the interpass holding time.

The multistage deformation behaviour of Al-5Mg-0.8Mn (5083) has been studied by McQueen et al. (1984) in the temperature range of 300-500°C with strain rates of 0.1 and 1 s\(^{-1}\) using torsion testing. Microstructural examination of this material revealed that both grain structure and substructure in quenched samples were similar for both continuous deformation and interrupted testing. These observations indicated that only static recovery occurred during intervals between passes, with the structure rapidly returning to its state prior to interruption. The ability to retain the old substructure was supported by the low level of fractional softening (10-35%), this was indicative of the effect of static recovery as reported by McQueen et al. (1984).

Recently, McQueen et al. (1989b) have examined microstructural development in Al-0.5Fe and Al-0.5Fe-0.5Co during multiple deformation. Multiple deformation was performed by torsion testing at temperatures of 400 and 450°C, at strain rates of 0.1 and 1 s\(^{-1}\), the delay time was varied from 5 to 100 sec. Optical microscopy of these alloys after multistage deformation exhibited serrated grain boundaries. The boundaries were more pronounced at higher temperatures, or longer delay times at lower strain rates, as a result of the more polygonized subgrains. Transmission electron microscopy of the samples given multipass deformation exhibited a more highly recovered substructure compared to samples given only single pass deformation. These observations suggested that static recovery had occurred mainly during the interpass hold time, no evidence of static recrystallization was found in these alloys.
1. 4. 3 Static Recovery and Static Recrystallization after Hot Deformation in Al and Its Alloys

Recovery and recrystallization occur during, after and between passes in metals and alloys subjected to high temperature deformation (McQueen and Jonas, 1985). The occurrence of static recovery and static recrystallization during holding time depends on stacking faults energy. In metals with high SFE such as aluminium, recovery occurs rapidly, while recrystallization is relatively slow. But in metals with low SFE, such as γ-Fe and copper, restoration occurs by recrystallization after initial static recovery during holding at the deformation temperature (McQueen and Jonas, 1975).

In aluminium and its alloys, because of the high level of dynamic recovery during deformation at elevated temperature, static recrystallization seldom occurs in intervals, or during air cooling after final deformation. The maximum softening by static recovery even after extended holding has been reported to be 60% in aluminium and its alloys (McQueen, 1991a). However, static recrystallization could take place on slow cooling or holding at the deformation temperature, but with a rate much slower than fcc metals.

Farag et al. (1968) have examined isothermal annealing of super-pure aluminium after hot working by simply holding, deformed samples simply held at the deformation temperature for different times prior to quenching. It was found that at high temperatures, 550-600°C, complete recrystallization occurred in short times, corresponding to the rest periods involved in commercial hot rolling of aluminium. At lower temperatures the time for recrystallization was longer than the rest periods, and relatively little change in microstructure occurred. Similar behaviour has been reported in Al-Mg alloys after hot working by Cotner and Tegart (1969). McQueen and Ryum (1985) have also reported similar results in aluminium and Al-Mg alloys. However, it was found that the time for
complete recrystallization in Al-Mg was longer than that for pure aluminium. This indicates that the presence of Mg slows down the rate of recrystallization.

Recently, Belling et al. (1990) have reported the absence of static recrystallization in 3004Al alloy on annealing at deformation temperature, even after 16 hours. However, static recrystallization did occur when the samples were held 50°C above the deformation temperature. This confirms that static recovery mainly takes place between passes, or during cooling in this material.

In summary, the following conclusions can be obtained from the above studies on multiple deformation in aluminium and its alloy.

1. The amount of fractional softening increases with increasing strain, strain rate, temperature and duration of intervals.

2. Static recovery occurs during interval in multistage deformation, because of the high level of dynamic recovery at high temperature deformation, however, there is insufficient strain energy available to nucleate static recrystallization of grains.

3. The presence of fine particles or precipitates in Al alloys decreases the amount of FS, because the fine precipitates stabilize the substructure by pinning boundaries.

4. Static recrystallization takes place by nucleation of grains within the network. Subgrains grow until the formation of a high-angle boundary which is capable of sustained growth until it impinges on some other boundary or is arrested at a precipitate.
1.5 Ductility and Fracture of Discontinuously Reinforced Aluminium MMCs

The use of metal matrix composites (MMCs) in structural applications is attractive because of their exceptional stiffness-to weight and strength to weight ratios. However, low ductility and fracture toughness of the composites remains a major obstacle to the practical application of these materials. Moreover, recently, there has been a growing interest in using these materials for high temperature applications (Park et al., 1990). It is thus important to examine the mechanisms that control ductility and fracture behaviour at low and high temperatures. The following section will deal with aspects of ductility and fracture at low and high temperatures in materials containing of second phase particles and MMCs.

1.5.1 Fracture of Materials Containing Second Phase

1.5.1.1 Fracture Behaviour in Materials with Low Volume Fraction of Particles

Numerous studies on fracture behaviour have been carried out on materials with low volume fraction of particles generally less than <0.1, as these materials best reflect the behaviour of many commercial alloys. It is well established that fracture in these materials is associated with void nucleation and growth, the nucleation of voids occurring at inhomogenities in the material such as; non-metallic inclusions, precipitates or artificially induced strengthening dispersions (Lindley et al., 1970; Ashby et al., 1978; Goods and Brown, 1979).
The role of particles in void nucleation at room temperature has been demonstrated by Puttick (1959) in polycrystalline copper containing small non-metallic inclusions. It was observed that voids nucleated at oxide inclusions either by decohesion at the particle/matrix interface or by inclusion cracking. This behaviour has been commonly observed in both ferrous and non-ferrous alloys. For example, Broek (1973) showed that voids may form at large particles, even at small strains, in aluminium alloys; and microcracks formed by particle cracking for long slender cracks. Palmer and Smith (1968) observed nucleation of voids after a strain of 0.1 in copper containing SiO₂ particles at room temperature. The results of Atkinson (1963) on this material with a volume fraction of SiO₂ four times less than that of Palmer and Smith, also showed that strain to nucleate voids was similar (Brown and Embury, 1973). These observations suggest that for a given particle size, the void nucleation strain does not depend on volume fraction alone.

Void nucleation can occur by decohesion at the interface between the particle and the matrix, and by cracking of the inclusions. During plastic deformation at room temperature a material containing hard particles disturbs the elastic and plastic displacement field in a deforming matrix. This disturbance leads to stress concentration at the particle. Plastic strain increases until the particle either separates from the matrix or fractures (Ashby et al., 1979). The deformation process occurring at the particle/matrix can thus be either particle fracture and/or interfacial decohesion, or plastic flow in the matrix during deformation in these materials.

Primary fracture models propose that voids nucleate at or near the particle/matrix interface by decohesion or particle-cracking, growth occurs by coalescence and finally the material fails. Basically these models propose that voids are nucleated at a critical nucleation strain. Once voids are nucleated, they grow under the applied stress and strain fields until they reach a critical size, and subsequently coalesce through failure of the matrix.
ligaments. This indicates that fracture in metals with low volume fraction of particles is controlled by void growth. Several models have been suggested for growth strain in materials containing particles with low volume fraction. The simple void growth model (Ashby et al., 1979) proposes that coalescence of voids will occur when the void length is equal to the interparticle spacing.

Brown and Embury (1973) have suggested that the coalescence of voids will occur when the void length is equal to the interparticle spacing. It is assumed that the voids are nucleated at a particle/matrix interface. The growth strain is equal to:

\[ \varepsilon_G = \frac{\pi}{6f} - \frac{2}{3} - 1 \]  

(1 - 30)

Evens and Verk (1981) have proposed a model to determine the growth strain when the voids are nucleated at a cracked particle. They developed the following equation for growth strain:

\[ \varepsilon_G = 0.104 \left( \frac{2\pi}{3f} \right)^{1/2} - \frac{8}{\sqrt{3}} \]  

(1 - 31)

Where \( f \) is volume fraction of particles. It can be seen from these models, that the growth strain depends only on the volume fraction of particles and is independent of particle size and the flow stress. In materials with a low volume fraction of second phase, these models show good agreement with experimental observations (Roy et al., 1981). It should be noted that these models are concentrated on the amount of growth strain required before the voids reach the critical size for linkage.

There are two criteria for a void to nucleate in a material containing particles (Hunt Jr. et al., 1987). The first and necessary condition for crack initiation is that the strain energy (\( U \)) stored at the inclusion is sufficient to provide the surface energy (\( S \)) of the newly formed crack surfaces, (or \( U > S \)). It has been shown (Tanaka et al., 1969) that this
energy criterion is almost satisfied for particles about 250Å but it is insufficient for nucleation of particles larger than 1 μm. Although the energy requirement is a necessary criteria for void nucleation, a local stress criteria must also be fulfilled. Local stress must be at least equal to the interfacial strength at the particle/matrix interface.

There are several factors which affect the void nucleation process such as matrix yield, work hardening, temperature and impurity, etc. The primary role of increasing yield strength is to activate a greater number of potential nucleation sites, by causing more particle matrix interfaces to reach their decohesion stress, or by increasing particle cracking. Work hardening behaviour has a similar effect on nucleation, stress in the vicinity of the particles is created with higher work hardening. But the role of work hardening in the void growth process is in contrast to void nucleation, where higher work hardening levels restrict the growth of stable voids and increases the strain to fracture. Another important factor to consider is the strength of the bond at the interface. The stronger this bond, the greater resistance to void nucleation.

The deformation temperature can also affect cavitation. At high temperatures, when dislocation mobility by climb is high, plastic relaxation is easy and local work-hardening is small. Also the rate of attaining the local stress could be decreased by allowing thermal or dynamic recovery. Under these conditions, nucleation of cavities is restricted (Goods and Brown, 1979). The volume fraction, distribution, shape and relative strength of the particles may all be of importance in relation to the ultimate ductility.

1.5.2 Fracture Behaviour in Materials with High Volume Fraction of Particles

In metals with high volume fraction of particles such as metal matrix composites, the fracture behaviour is different from that observed in materials with low volume fraction of
particles. The volume fraction of particles influences the nucleation and growth strain. Increasing the volume fraction of particles leads to an overlap of plastic zones and a decrease in nucleation strain, the interparticle spacing decreases also with increasing volume fraction resulting in decreasing of the growth strain. Although some investigators have observed that voids can form and remain stable in Al-10%vol Al₂O₃, while straining continues (Whitehouse et al., 1991), the extensive experimental evidence suggests that occurrence of stable voids in commercial MMCs is rare (Hunt Jr. et al., 1987). This is because the majority of damage process is confined to regions very close to final fracture surface and no stable voids are seen in MMCs. These observations indicate that the process of void nucleation seems to be the critical process in the fracture of materials with particle volume fraction greater than ~ 0.1.

Comparison of the fracture process in metals with low and high volume fraction indicates that the void nucleation process is the similar. However, in the low volume fraction materials, void growth plays a critical role, while the void nucleation is critical in metals with high volume fraction of particles. The possibility of particle clustering is much greater when a high volume fraction of particles is present due to the reduction of interparticle spacing. It is known that damage can occur preferentially in regions of low interparticle spacing, this can lead to a percolation of damage through clustered regions of high local volume fraction. As the volume fraction of hard particles is increased these effects become of major importance; initial decohesion events become dominant and fracture occurs by the percolation of damage, prior to any significant macroscopic strain (Embury, 1985).
1.5.2 Fracture of Discontinuously Reinforced Aluminium

MMC at Room Temperature

The limited ductility of most discontinuously reinforced MMCs is thought to be due to, at least in part, a tendency for cavities to be nucleated at reinforcing particles or fibers, and subsequently for growth and linkage to promote final failure (Logsdon and Liaw, 1986). During room temperature deformation, very large elastic stresses are built up in the reinforcement as a result of the incompatibility of the particles and matrix. This induces matrix yield and plastic deformation before interfacial debonding, and in some cases these stresses cause the particles to fracture (Liu et al., 1991). It has been established from fracture studies that particles in the larger size range (>10 μm) are more likely to fracture than smaller particles. Particle fracture is also expected to be more severe in stronger composites as the fracture of ceramic reinforcement is stress rather strain dependent as pointed out by Llyod (1989). This means that particle fracture is a function of matrix composition and particle volume fraction.

As mentioned in the previous section, in materials with high volume fraction such as SiC/Al composites, the interparticle spacing decreases and the amount of growth strain required also decreases (equations 1-30 and 1-31). Assuming a uniform particle size and distribution, it can be estimated as the volume fraction increases to about 0.16 the amount of growth strain becomes zero. Experimental and theoretical work suggests that void nucleation is critical in materials with a high volume fraction of particles. Particle clustering can also play a significant role in the fracture initiation process in high volume fraction materials, such as SiC/Al composites.

McDanel's (1985) studied the effect of volume fraction of SiC whiskers on ductility and fracture behaviour of 6061Al/SiCw composite. It was found that the ductility decreased
with increasing reinforcement content, and composites with low volume fraction of reinforcement (10 to 15%) exhibited the same fracture surface with wrought alloy. However, the composite with 30 to 40% Vol. of SiC showed cleavage. Flom and Arsenault (1987) found that the fracture process in SiCp/1100Al composite was matrix controlled for SiC particles less than 20 μm, however, when the size exceeded 20 μm the particle fracture dominated. Fracture surfaces revealed that the composite exhibited dimple morphology, being a typical process of ductile fracture of matrix with two different dimple sizes. The first one was associated with SiC particles where large dimples were present, and the other were the very fine dimples located in the space between the particles. Particle fracture was not observed for small particle sizes (< 10 μm). The shape of the dimples was rather equiaxed and no voids observed below the fracture path, indicating that the fracture process was confined to an extremely narrow band. There was also no apparent damage zone adjacent to the fracture surface which generally exists in the classical void nucleation and growth type in ductile fracture. Similar dependence of the fracture process on particle size has been reported by Shyong and Ruiz (1993) in 6061Al/SiCp composite. It was found that the failure modes changed from cracking of the particle/matrix interface to decohesion at the particle/matrix interface where particle size decreased from 30 to 3 μm.

The effect of matrix microstructure and particle size on fracture behaviour of 7091Al/SiCp composite produced by powder metallurgy has been studied by Lewandowski et al. (1988), Manoharan and Lewandowski (1989; 1990; 1992) and Lewandowski and Liu (1989). In their studies, two different heat treatments were used to produce underaged* and overaged* in the matrix and two different particle size, 5 and 13 μm.
Chapter One Literature Review

Their results showed that the fracture tended to initiate in clustered regions, regardless of matrix temper. It was found that fracture of the SiC particles was predominate in the underaged composites, as the temper varied from the underaged to overaged condition decohesion became the predominate fracture mode. The reason for this transition seems to be due to the presence of precipitates at the particle/matrix interface. The precipitates reducing the interface strength and resulting in an increase in particle/matrix decohesion for the overaged condition, irrespective of particle size. Although the above results indicate a pronounced effect of heat treatment on fracture mode in 7xxxAl/SiCp composites, a minor effect of matrix microstructure on the fracture behaviour has been observed in 2xxxAl/SiCp composites. For example, You et al. (1987) found a similar fracture behaviour in the 2124Al/20%SiCp composite in both T4** and overaged** conditions.

In summary, in particulate composites fracture initiation is associated with either particle fracture or interfacial-matrix failure, being dependent on the reinforcement size and matrix condition or both (Llyod, 1991; Hunt Jr. et al., 1993). Preferential void nucleation has been observed in regions of high volume fraction of particles or regions of particle clustering. Particle fracture has been observed, particularly for large particles (> 10 μm). Void nucleation occurs at a particle/matrix interface by either cracking of the particle or decohesion at the interface. The fracture surface of MMCs exhibits dimpled, and cup and cone appearance similar to that exhibited by monolithic alloys.

* Solution treated at 500°C for 4 hours and quenched in water, then aged at 120°C for 20 minutes to obtain underaged and aged at 120°C for 24 hours followed by holding at 170°C for 36 hours produced overaged condition.

** Solution treated at 495°C for 1 hour followed by cold water quenching and naturally aged at room temperature for 48 hour (T4) and to obtain overaged the samples held at 190°C for 24 hours.
Chapter One  Literature Review

1. 5. 3  Ductility and Fracture of Discontinuously Reinforced Aluminium MMCs at High Temperatures

Fracture behaviour and mechanical properties of metal matrix composites based on 6061, 2124, 7090 and A356 aluminium reinforced with whiskers and particulate SiC have been studied by several investigators. Sakaris and McQueen (1992a; 1992b) and McQueen et al. (1993) have investigated the hot ductility of A356Al/15%SiCp and 6061Al/15%SiCp composites under conditions of torsion loading. It was found that ductility of both composites and matrix alloys increased as deformation temperature increased. The SiCp/A356 composite showed much higher $\varepsilon_f$ compared to the SiCp/6061Al composite. In fact, $\varepsilon_f$ in SiCp/A356Al composite reached a maximum of 4 above 400°C at a strain rate of 0.1s$^{-1}$ but $\varepsilon_f$ of the composite was ~ 20 to 30% less than the matrix. The results showed that the ductility for the composite increased up to 500°C at various strain rates and remained almost constant up to melting temperature.

Pickens et al. (1987) found that 7090/SiC$_w$ composite fractured at a strain of 0.6 at 427°C at a strain rate of 0.1s$^{-1}$, whereas monolithic 7090 aluminium matrix exhibited much higher fracture strain. The ductility of 6061Al/SiC$_w$ also increased with increasing deformation temperature up to 427°C and remained nearly constant up to melting temperature. The ductility increased with increasing of strain rate for both composites in the range of $10^{-3} - 10^{-1}$ s$^{-1}$, at which point it remained constant for the 6061/SiCw while decreased for 7090/SiCw. While Chanda et al (1991) did not find a strong strain rate dependence of tensile ductility in Al-4Li alloy reinforced with SiC particles, in the range of strain rates of $10^{-5}$ to $10^{-1}$ s$^{-1}$ at 525°C.
The creep behaviour of 6061 aluminium alloy reinforced with silicon carbide whiskers and particulate has been studied by Nieh et al. (1988a) at temperatures up to 370°C. The SEM fracture surfaces showed ductile failure in these materials. The large amount of plastic deformation that occurred in the matrix suggesting that the composites fractured by coalescence of voids developed in the matrix during creep deformation. It was found that most of dimples associated with the SiCw indicating the interface of SiCw/Al to be the preferential sites for cavity initiation. Both types of composites (whisker and particulate) failed by plastic tearing of the aluminium matrix. A similar fracture behaviour in aluminium based MMCs under creep conditions has been reported by other investigators (Nardone and strife, 1987; Gungor et al., 1988; Park et al., 1990)

Results from studies on tensile behaviour of aluminium based MMCs at high temperatures show that the tensile strength of these materials decrease as temperature increases, but at higher temperatures the composite and the matrix alloy show a similar strength levels. However, the composites show much lower ductility compared to the matrix alloys. The fracture surfaces of specimens deformed at high temperatures were generally found to be similar to those at lower temperatures. The fracture mechanism at high temperatures was found to be due to nucleation of voids at the matrix/particle interface and inclusions. The composites exhibited some external necking prior to fracture at high temperatures (Philips, 1987; Almas and Humphreys, 1988), while the matrix alloy shows different behaviour.

Recently, Zhao et al. (1994) have studied the tensile behaviour of 2014 aluminium alloy MMC, reinforced with 10, 15 and 20 %vol. of Al2O3 particles, as a function of temperature between 100 and 300°C. Their results showed that at lower temperature up to 200°C, the composites exhibited higher yield strength and UTS than the unreinforced alloy. But at higher temperatures, all materials showed similar strength levels. Elongation increased with increasing temperature but was lower than the unreinforced
alloy. It was found that at lower temperatures particle fracture was the main damage prior to final fracture, whereas interparticle voiding was dominant at higher temperatures. Void nucleation was found in the matrix between closely particles and particle ends and the amount of void increased as temperature increased. Similar results have been reported in 2014Al/20%SiCp composite by Corbin and Wilkinson (1989).
CHAPTER 2
Materials and Experiments
2.1 Materials

This chapter describes the materials used in this work, the preparation of the specimens as well as the techniques utilized for testing and examining them. The materials used in this work were 7075 aluminium monolithic alloy and a composite which contained 15% volume fraction of silicon carbide (SiC) particles in 7075 matrix. The average particle size of SiC was 14 μm. Both materials were supplied by Alcan Canada Ltd in the form of hot extruded rectangular bars. These materials were made by ingot metallurgy. Composition limits of 7075 aluminium alloy are shown in Table 2.1.

Table 2.1 Composition limits of 7075 aluminium alloy (wt %)

<table>
<thead>
<tr>
<th></th>
<th>Zn</th>
<th>Mg</th>
<th>Cu</th>
<th>Cr</th>
<th>Mn</th>
<th>Si</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>5.1 - 6.1</td>
<td>2.1 - 2.9</td>
<td>1.2 - 2.0</td>
<td>0.18 - 0.28</td>
<td>0.8 max</td>
<td>0.4 max</td>
</tr>
<tr>
<td>Fe</td>
<td>0.5 max</td>
<td>0.2 max</td>
<td>Others (each)</td>
<td>Others (total)</td>
<td>A 1</td>
<td>Balance</td>
</tr>
<tr>
<td>Ti</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Others</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2.1.1 Microstructure: As-received Materials

The optical micrograph of as-received 7075Al/SiCp composite is shown in Fig. 2.1. It can be seen that the distribution of SiC particles in the matrix is not very uniform and the clusters of ceramic particles can be observed in some regions. The particles are also aligned in the extrusion direction. The optical microstructure of 7075 aluminium monolithic alloy in the as-received condition is shown in Fig. 2.2 which clearly shows elongated grains in the direction of extrusion. A high dislocation density is observed.
Extrusion direction

Fig 2.1 Optical micrograph of as-received 7075Al/SiCp composite

Figure 2.2 Optical micrograph of 7075Al monolithic alloy in as-received condition
around the particles in the reinforced alloy as shown in the transmission electron micrograph, Fig. 2.3. This high dislocation density is generated either by deformation during hot extrusion or during cooling as a result of the mismatch in the coefficient of thermal expansion (CTE) of the matrix and SiC particles. Figure 2.4 shows the morphology of the SiC particles with irregular shape and rough surface along with some fractured particles in the matrix.

2.1.2 Heat Treatment

Since the materials in as-received condition were in unrecrystallized state, in order to obtain materials in the fully recrystallized state, both the reinforced and unreinforced alloys were annealed and then cold rolled and subsequently annealed again to achieve the desired structure. The materials were annealed at 425°C for 2.5 hours and cooled slowly in the furnace followed by holding at 230°C for 2.5 hours and then air cooled. The microstructure of 7075Al alloy after annealing generally consists of both coarse and fine precipitates of (MgCu)Zn$_2$ phase, the dispersoids Al$_{18}$Mg$_{23}$Cr$_2$ and constituent phases Al$_7$Cu$_2$Fe, (Al,Cu)$_6$(Fe,Cu), Mg$_2$Si, $\alpha$-Al$_{12}$Fe$_3$Si (Raghavan et al., 1985). It should be noted that these materials were tested in tension in both annealed and T6 conditions. In order to achieve T6 condition, the tensile specimens were first solution treated at 480°C for 2.5 hours, quenched in water and then aged at 120°C for 24 hours.

2.1.3 Hot Compression and Tensile Specimens

The samples for single and double pass deformation were machined in form of cylindrical shape of 8 mm in diameter and 12 mm in length and the cylinders were prepared parallel to the extrusion direction. Nickel anti-seize tape was used as lubricant to minimize bulging of the samples during compression at high temperatures. For the tensile tests, the cylindrical specimens (Hounsfield No. 12) with a 16 mm in gauge length and 4.5 mm
Figure 2.3 TEM micrograph of the as-received 7075Al/SiCp composite showing high dislocation density in vicinity of particle

Figure 2.4 The morphology of the SiC particulate in the composite
diameter were machined from the as-received materials. The dimensions of tensile and compression specimens are shown in Fig 2.5 (a-b).

2. 2 Mechanical Testing

2. 2. 1 Hot Compression Testing

Two types of compression tests were conducted in this work; single pass and double pass deformation at high temperature. For the single pass experiments, the uniaxial compression testing was carried out by using a modified servo-hydraulic Instron testing machine with a radiation infrared furnace attachment. The compression machine was linked to a computer so that strain rate, strain and temperature could be controlled. In addition, the computer was used to collect the relevant data for plotting true stress-true strain curves. Compression tests were carried out in the temperature range from 250 to 450°C at constant strain rates from 0.001 to 1 s⁻¹. Prior to testing, the cylindrical samples were heated up to the test temperature, held for 10 minutes and then compressed to a true strain of 0.9. The samples were quenched immediately after deformation into water for microstructural investigation.

For double pass deformation study, the samples were first compressed to a true strain of 0.4 in the first pass, held at the deformation temperature for different delay times ranging from 6 to 900 sec and then compressed to a total true strain of 0.9. The double pass deformation tests were carried out at 300 and 400°C at a constant strain rate of 1 s⁻¹. To study the microstructural changes occurring during holding time, a series of samples were deformed to a true strain of 0.4, held at the test temperature for different times, and then quenched. The Schematic of the deformation schedule used in compression experiments is shown in Fig. 2.6.
Figure 2.5 Schematic shape and dimensions of samples a) tensile sample, b) compression sample
Figure 2.6  Schematic diagram of hot compression deformation a) single pass,  
b) double passes
The effect of annealing temperature on the static recrystallization of reinforced alloy after hot working was studied. The samples were deformed to a true strain of 0.9 in the temperature range of 250-400°C at a strain rates of 1 s\(^{-1}\) and then annealed at deformation temperature as well as above deformation temperature for 1 hour and quenched. A series of samples deformed at strain rates of 0.01, 0.1 and 1 s\(^{-1}\) between 250-400°C were annealed at 500°C for 1 hour to study the effect of strain rate on the extent of static recrystallization in the reinforced alloy.

2. 2. 2 High Temperature Tensile Testing

Tensile tests were carried out on a servo-hydraulic Instron machine under constant strain rate conditions. The machine was equipped with an IBM compatible computer to control the strain rate, acquire the data, as well as monitor the load against displacement. Hounsfield type round tensile samples were machined from the as-received materials and heat treated to produce annealed and T6 conditions. The elevated temperature tests were carried out within a three-zone vertical furnace attached to the tensile machine. The tests were conducted in a temperature range from 25 to 400°C at a strain rate of 0.001 s\(^{-1}\). A series of samples were tested at 400°C at strain rates of 0.001, 0.01 and 0.1 s\(^{-1}\) to study the effect of strain rate on tensile ductility and fracture behaviour of the composite. Prior to testing, the samples were first heated to the test temperature, held at that temperature for 10 minutes to obtain uniform temperature and then strained in tension. The specimen temperature was controlled by a chromel-alumel thermocouple placed in contact with the specimen within the gauge length. All the tensile samples were pulled to fracture. The specimens were quenched following the test by blasting liquid CO\(_2\) onto the samples held in the grips. The tensile ductility was measured as a percentage of elongation of the samples after fracture. The hot compression and tensile machine equipment are shown in Fig. 2.7.
Figure 2.7 Computer aided servo-hydraulic testing machines a) Compression equipment b) Tensile machine
2. 3 Metallographic Techniques

2. 3. 1 Optical Metallography

Optical metallography was used for microstructural examination and grain size measurement of the composite and the matrix alloy. The examinations were performed on the samples prior to and subsequent to deformation, at different temperatures and strain rates. In addition, a further examination was made to study the longitudinal sections of fractured specimens. The grain size measurement was undertaken by linear intercept method.

The 7075 aluminium monolithic alloy specimens were cut from the deformed samples, then prepared for metallographic examination by first grinding through successively finer grades of SiC papers of mesh size of 180, 240, 320, 400, 600 and finally polishing on soft cloth impregnated with 6 and 1 micron diamond spray, respectively. In the case of the 7075Al/SiCp composite, the samples were first ground down with SiC papers of mesh size 120, 240, 320, 400, 600, respectively. They were then polished on soft cloth followed by polishing on Textmet cloth impregnated with 6 and 1 micron diamond spray for 3 minutes. Final polishing was conducted on Microcloth carrying colloidal silica and water to remove scratches on matrix area in the composite. Modified Keller's reagent was used for etching and the composition of the etching solution was, 2 ml HF, 3 ml HCL, 20 ml HNO₃ and 175 ml H₂O. The samples were etched by immersing in the solution for 60 seconds and then washed in stream of warm water and finally dried by blowing. The grain size measurement was carried out by the linear intercept method in which at least 200 grains at ten random areas per sample were taken and the average grain size was estimated.
2. 3. 2 Quantitative Image Analysis

An image analysis computer was used to determine the volume fraction and particle size of SiC particles in the composite as well as to estimate the volume fraction of fractured particles on the fracture surface. The volume fraction of SiC particles in 20 random areas in a polished sample was measured and the average volume fraction of particles in the composite was calculated. The volume fraction of ceramic particles in the composite was ~15%. The mean particle size of SiC was estimated by the measurement of at least 400 particles and the distribution of particle size is shown in Fig. 2. 8. It can be seen that 70% of SiC particles present have a size between 10 to 15 µm whilst the average particle size of SiC was estimated to be 14 µm.

2. 3. 3 Electron Microscopy

(a) Scanning Electron Microscopy

A LECO STEREOSCAN 440 scanning electron microscope was used to examine the fracture surfaces, longitudinal sections of fractured specimens, as well as morphology of SiC particles in the composite. The fracture surfaces were examined for the details of fracture process, whereas polished longitudinal sections were observed for detection of cavitation as well as damage mechanisms in the composite. Polished samples were first shadowed with gold by vacuum coating to obtain a good resolution and depth of focus. The back scatter electron was used to distinguish the particles and second phase precipitates in the matrix. The energy dispersive spectroscopy x ray (EDSX) was also used to analyse the surface of SiC particles on the fracture surface as well as precipitates.
Figure 2.8 The distribution of particle size in 7075Al/SiCp composite
The main advantage in using SEM for these observations is the greater depth of focus which can be obtained at high magnification as well as to obtain x ray analysis.

(b) Transmission Electron Microscopy

Transmission electron microscopy has been utilized to study the microstructural development such as substructure, dislocation network and precipitates in the deformed samples. Following is the procedure adopted for the preparation of thin foils for TEM work:

1. cutting the slices with a thickness of about 1 mm with a Vericut machine, the slices were cut parallel to the direction of deformation axis of cylindrical samples.

2. punch out 3 mm diameters discs by spark machine

3. grinding the discs to a thickness of 0.1 to 0.15 mm by 600 micron SiC paper, and

4. electropolishing.

Electropolishing was performed using a Struers Tenupol polisher in a solution of 25 parts of nitric acid and 75 parts of methanol at a temperature of -30°C at a voltage of 30V.

The subgrain size in the deformed samples was measured on the TEM photomicrographs taken from at least two thin foils and at least 5 areas from each sample.

The transmission electron microscopy was carried out using a JEOL 2000 FX operating at 200 KV.
CHAPTER 3

Deformation Behaviour and Microstructural Development at High Temperature
3.1 Introduction

Discontinuously reinforced aluminium metal matrix composites can be produced either by primary fabrication processes such as ingot metallurgy techniques, and casting, or by powder metallurgy and they are commercially available in large quantities suitable for many structural applications. One of the prime advantages of the aluminium based MMCs is that the billets of the composites can be fabricated by conventional metalworking processes such as extrusion and rolling. Deformation processing of these materials is usually carried out at high temperatures where the ductility is high and the flow stress is relatively lower. It is thus necessary to understand the deformation behaviour and microstructural development in these materials at elevated temperature. In this chapter, the effect of temperature and strain rate on the flow behaviour, strength and microstructure of the 7075Al monolithic alloy and a 7075Al/SiCp composite will be presented based on experimental results.

The hot deformation behaviour was studied under uniaxial compression in the temperature range from 250 to 450°C at strain rates from 0.001 to 1 s⁻¹. After deformation, the samples were quenched in water immediately for microstructural investigation.

3.2 Results

The high temperature deformation study was carried out for both unreinforced and reinforced 7075Al alloy in fully annealed condition. The initial microstructures of the composite and the matrix alloy prior to deformation are shown in Fig. 3.1 (a-b). It can be seen that these materials exhibit elongated grains parallel to the direction of extrusion. The initial grain size was ~ 14.5 and ~22.5 μm for 7075Al /SiCp composite and 7075Al alloy, respectively.
Figure 3.1 Initial microstructure of the materials prior to compression test
   a) 7075Al/SiCp composite and b) 7075 Al alloy
3. 2. 1 Flow Behaviour Characteristics

True stress-true strain curves of 7075Al matrix alloy and 7075Al/SiCp composite in the temperature range of 250-450°C at constant strain rate of 0.1 and 1 s⁻¹ are shown in Figs. 3.2 and 3.3, respectively. It can be seen that the flow stress rises rapidly, attains maximum level of stress at low strain and then reaches a steady state at higher strain. The comparison of the flow curves of the reinforced and unreinforced alloys shows that the former exhibits higher flow stress level compared to the monolithic alloy at temperatures up to 400°C. The flow curves are typical of the form in metals which show dynamic recovery during deformation at high temperatures. It can also be seen from the flow curves that at a given strain rate, the flow stress decreases with increasing temperature and decreases as strain rate decreases at a given deformation temperature. A real curve obtained from the computer attached to compression equipment contains some noise signals, Fig. 3.4, which arise probably from testing machine and control system. However, the noise has no effect on true flow behaviour of the materials under different conditions of strain rate and temperature.

3. 2. 2 Effect of Strain Rate and Temperature on the Flow Stress

The relationship between flow stress and strain rate at a constant temperature can be expressed by the empirical relation by following equation:

\[ \sigma = K \dot{\varepsilon}^m \]  \hspace{1cm} (3 - 1)

Where \( \sigma \) is flow stress, \( \dot{\varepsilon} \) is strain rate, \( K \) is a constant and \( m \) is the strain rate sensitivity which is the slope of the plot \( \ln \sigma \) vs \( \ln \dot{\varepsilon} \). The stress dependence on strain rate can be
Figure 3.2 True stress - true strain curves of 7075Al alloy at different temperatures at constant strain rates of a) 1 s$^{-1}$, b) 0.1 s$^{-1}$.
Fig 3.3  True stress-true strain curves of 7075Al/SiCp composite at different temperatures at constant strain rates of a) 1 s⁻¹, b) 0.1 s⁻¹
Figure 3.4 The real curve for 7075Al/SiCp composite at high temperature which contains some noise signal from the testing machine and/or control system.
obtained from the plots of $\ln \sigma$ vs $\ln \dot{\varepsilon}$ for the composite and the matrix alloy shown in Fig. 3.5. It can be seen that both materials show linear curves. The strain rate sensitivity, $m$ can be measured from the slope of $\ln \sigma$ vs $\ln \dot{\varepsilon}$ and Table 3.1 shows the $m$ values of the unreinforced and reinforced alloys at different temperatures. It can be seen that the composite displays a slightly higher $m$ value compared to the monolithic alloy under identical conditions.

Table 3.1  $m$ values for 7075Al alloy and 7075Al/SiCp composite

<table>
<thead>
<tr>
<th>Temperature ($^\circ$C)</th>
<th>7075Al</th>
<th>7075Al/SiCp</th>
</tr>
</thead>
<tbody>
<tr>
<td>250</td>
<td>0.076</td>
<td>0.089</td>
</tr>
<tr>
<td>300</td>
<td>0.089</td>
<td>0.1</td>
</tr>
<tr>
<td>350</td>
<td>0.11</td>
<td>0.13</td>
</tr>
<tr>
<td>400</td>
<td>0.138</td>
<td>0.153</td>
</tr>
<tr>
<td>450</td>
<td>0.21</td>
<td>0.23</td>
</tr>
</tbody>
</table>

The combined effect of strain rate and temperature on flow stress under hot working conditions can be described by using Zener-Holloman parameter according to the following relation:

$$Z = \dot{\varepsilon} \exp\left(-\frac{Q}{RT}\right) = A \left[\sinh\left(\alpha \sigma\right)\right]^n$$  \hspace{1cm} (3-2)

Where $A$, $\alpha$ and $R$ are constants which are independent of temperature and stress, $\dot{\varepsilon}$ is constant strain rate, $\sigma$ is flow stress (maximum true stress) and $T$ is deformation temperature (in Kelvin), $n$ is stress exponent and $Q$ is activation energy for plastic flow. It can be seen that to plot the variation of $Z$ with $\sigma$ the values of $\alpha$, $n$ and $Q$ must be known.
Figure 3.5  Plots of $\ln \sigma$ vs $\ln \dot{\varepsilon}$ for a) 7075Al/SiCp composite and b) 7075Al alloy at different temperatures
Since values of $\alpha$ and $n$ for the composite and the matrix alloys were not available, the method proposed by Uvira and Jonas (1968) was used to calculate these values. $\alpha$ can be taken to be equal to $\beta/n_1$ (Dieter, 1976) and $\beta$ can be obtained from the slope of the $\ln \dot{\varepsilon}$ vs $\sigma$ curves according to the following equation:

$$\dot{\varepsilon} = A_1 \exp (\beta \sigma) \tag{3-3}$$

$n_1$ is equal to $1/m$, which $m$ is strain rate sensitivity. $n$ value was determined from the following relation:

$$n = \left[ \frac{\delta \ln \dot{\varepsilon}}{\delta \ln \sinh (\alpha \sigma)} \right] \tag{3-4}$$

which was obtained from Eq. 3.2 in assuming that the activation energy and the parameter $A$ were constant over the range of strain rate and temperature used in this study. The most suitable value of $\alpha$ was the one which led to the minimum standard deviation in $n$ over the full range of temperature studied. The $\alpha$ value was found to be 0.027 and 0.058 for the reinforced and unreinforced alloy, respectively. Fig. 3.6 shows the plot of $\ln \dot{\varepsilon}$ as a function of $\ln [\sinh (\alpha \sigma)]$ for the composite and the matrix alloy, respectively.

It can be seen that the data fit well and the relationship between flow stress, strain rate and temperature in these materials follow above relation in equation 3.2. The stress exponent, $n$ value calculated were estimated to be 3.28 for the reinforced alloy and 2.02 for the monolithic alloy.

The activation energy for both materials was evaluated from the plots of $\ln (\alpha \sigma)$ vs $1/T$ at different strain rates, as shown in Fig. 3.7a and b. $Q$ values were evaluated from the slope of these lines and was found to be 152 kJmole$^{-1}$ for the monolithic alloy and
Figure 3.6  Plots of $\ln \dot{\varepsilon}$ vs $\ln \sinh (\alpha \sigma)$ for a) 7075Al/SiCp composite and b) 7075Al alloy at different temperatures
Chapter Three

Figure 3.7 Plots of \( \ln \sinh (\alpha \sigma) \) as a function of \( 1/T \) at different strain rates for a) 7075Al/SiCp composite and b) 7075Al alloy, activation energy for deformation, \( Q \), was evaluated from the slope of the lines.
168 kJmole\(^{-1}\) for the composite material. The activation energy of the composite is slightly higher than that of the monolithic alloy.

With calculating \(\alpha\), \(n\) and \(Q\), the temperature and strain rate dependence of flow stress can be expressed by the variation of \(\ln Z\) vs \(\ln [\sinh (\alpha \sigma)]\) as shown in Fig. 3.8 for the reinforced and unreinforced alloys. It can be seen that as \(Z\) increases, i.e. temperature decreases or strain rate increases, flow stress increases in both materials.

### 3.2.3 Effect of Temperature and Strain Rate on the Yield Stress

The flow curves show that the reinforced and unreinforced alloys do not exhibit sharp, well defined yield point. The stress corresponding to 0.2% plastic strain was taken as yield stress. The variation of 0.2% yield stress as a function of deformation temperature at various strain rates are shown in Fig. 3.9. It can be seen that the yield stress in both monolithic and reinforced materials decreases gradually as deformation temperature increases. Figure 3. 10 shows a linear relationship between 0.2% yield stress and strain rate on ln-ln scale, which clearly indicates that the yield stress decreases as strain rate decreases. At low temperatures (250 and 300°C) the yield stress decreased slightly as strain rate increased but at high temperatures (400-450°C) both materials showed a much pronounced strain rate dependence of yield stress.

### 3.2.4 Effect of Temperature and Strain Rate on Strain Hardening

Fig. 3.11 shows the strain hardening rate as a function of temperature for the composite and the monolithic alloy. It can be seen that strain hardening rate decreases as temperature increases in both materials. The effect of strain rate on strain hardening rate is shown in Fig. 3.12 in the temperature range from 250 to 450°C. It was found that strain hardening
Figure 3.8 Flow stresses are related to temperature compensated strain rate through a plot of $\ln Z$ against $\ln [\sinh (\alpha \sigma)]$ for a) 7075Al/SiCp composite and b) 7075Al alloy
Figure 3.9 The Variation of yield stress as a function of temperature at various strain rates for (a) 7075Al/SiCp composite and (b) 7075Al alloy
Figure 3.10  Plots of 0.2% yield stress as a function of strain rates at different temperatures for a) composite and b) monolithic alloy
Figure 3.11  Strain hardening as a function of deformation temperature at various strain rates for a) composite and b) monolithic alloy
Figure 3.12 Effect of strain rate on initial strain hardening rate at different temperatures for a) the composite and b) the monolithic alloy.
rate decreased as strain rate decreased and at lower temperatures, the composite had a higher strain hardening rate compared to the monolithic alloy, but as temperature increased or strain rate decreased the difference in the strain hardening rate in both materials decreased. At 450°C and low strain rate both materials exhibited the similar strain hardening rate.

3. 2. 5 Microstructural Development During Hot Compression in 7075Al/SiCp Composite

3. 2. 5. 1 Optical Microscopy

Figure 3.13 shows the microstructures of the composite deformed in the temperature range from 300 to 450°C at a strain rate of 1 s⁻¹. The elongated grains in the direction normal to compression axis are present in these samples deformed at the different temperatures. The grain boundaries can not be seen in sample deformed at 300°C as a result of shearing, but as temperature increases the grain boundaries become observable and a few equiaxed grains along with serrated elongated grains can be seen in sample deformed at 450°C.

Optical microstructures of 7075Al/SiCp composite deformed at 400°C at strain rates of 0.1 and 0.01 s⁻¹ are shown in Fig. 3.14. Both samples show elongated grains but the grains in sample deformed at lower strain rate is coarser compared to one deformed at higher strain rate. Similar microstructures were observed in monolithic alloy deformed under identical conditions, however, the grains were coarser than that in the composite.
Figure 3.13  Optical micrographs of 7075Al/SiCp composite deformed at a constant strain rate of 1 s\(^{-1}\) a) 300°C  b) 350°C  c) 400°C  d) 450°C
Figure 3.14  Optical micrographs of 7075Al/SiCp composite deformed at 400°C at strain rates of (a) 0.1 s\(^{-1}\) and (b) 0.01 s\(^{-1}\).
3.2.5.2 Transmission Electron Microscopy

TEM micrographs of deformed 7075Al/SiCp samples at different temperatures in the range of 250 to 450°C at a constant strain rate of 1 s\(^{-1}\) are shown in Fig. 3.15. The sample deformed at 250°C shows a cellular character with high dislocation density and fine precipitates within the cells as well as on the cell walls. The subboundaries can hardly to be observed at 250°C, but as temperature increases, the subgrain size increases and subboundaries become well-defined due to increasing degree of dynamic recovery and coarsening of precipitates. An equiaxed subgrains can clearly be seen in sample deformed at 450°C. Figure 3.16 shows the substructure developed in the composite in the temperature range 300 to 450°C at a strain rate of 0.1 s\(^{-1}\). The presence of substructure is observed at all temperature and the subgrains are relatively fine at 300°C. But as temperature increases to 450°C the subgrains become coarser and the subboundaries become sharper. Within these large subgrains with well defined boundaries a significant dislocations can be observed at high temperatures (400 and 450°C). A smaller substructure can be seen in the samples deformed at 1 s\(^{-1}\) in comparison to the those deformed at 0.1 s\(^{-1}\) at same temperature. The presence of precipitates inside subgrains and subboundaries was found in all samples deformed under different temperature and strain rate conditions. The TEM micrographs of the monolithic alloy deformed in temperature range of 300-450°C at strain rate of 0.1 s\(^{-1}\), Figure 3.17, also exhibit increasing of subgrain size as temperature increases.

The effect of strain rate on the substructural development in 7075Al/SiCp composite at 400°C is shown in Fig. 3.18. It can be seen that the equiaxed subgrains are present at all strain rates, however, the subgrain size increases as strain rate decreases. The average subgrain size at highest strain rate of 1 s\(^{-1}\) was 1.8 μm whilst much larger size over 4 μm was obtained at 0.001s\(^{-1}\) at 400°C. The monolithic alloy also exhibited similar behaviour.
Figure 3.15 TEM microstructures of 7075Al/SiCp composite deformed at a constant strain rate of 1 s\(^{-1}\) at a) 250°C b) 300°C c) 350°C d) 450°C
Figure 3.16  TEM micrographs of 7075Al/SiCp composite deformed at strain rate of 0.1 s\(^{-1}\) a) 300°C b) 350°C c) 400°C d) 450°C
**Figure 3.17** TEM microstructures of 7075Al deformed at a strain rate of 0.1 s\(^{-1}\) at a) 300°C b) 350°C c) 400°C d) 450°C
Figure 3.18  TEM microstructures of 7075Al/SiCp composite deformed at 400°C at
a) 1 s\(^{-1}\) b) 0.1 s\(^{-1}\) c) 0.01 s\(^{-1}\) and d) 0.001 s\(^{-1}\)
The average subgrain size in the composite and the monolithic alloy at different temperatures and strain rates are given in Tables 3.2 and 3.3. The results show that the subgrain size increases as temperature increases and strain rate decreases in both reinforced and unreinforced alloys. It can be seen that the composite shows finer substructure in comparison to the monolithic alloy at lower temperatures and higher strain rates. But as temperature increases and strain rate decreases, the difference in subgrain size between reinforced and unreinforced alloy decreases.

3.2.5.3 A Microanalysis Investigation

TEM Selective Area Diffraction Pattern (SADP) technique was used for microanalysis of the composite samples deformed at different temperatures and strain rates. Figure 3.19 shows the TEM micrograph of the composite deformed at 300°C, 1 s⁻¹, with diffraction pattern taken from each grains. The same diffraction pattern and zone axis, [013], in the grains A, B, C, D and E indicate that the misorientation of boundaries between these grains is small and they can considered as subboundaries. But the zone axis in the grain F is [001], which is different from other grains. This is indicative of high angle boundary between the grains F and A. It should be noted that the grain F is a small part of an elongated grain which consists of substructure. This means that this grain boundary can be an original grain boundary but not new grain formed during deformation.

A similar feature is found in the sample deformed at 400°C, 1 s⁻¹, as shown in Fig 3.20. The SADP from an area consisting of several grains A, B, C, D, and E shows the same set of diffraction patterns with a zone axis of [011]. The same diffraction pattern and zone axis obtained is indicative of low misorientation between these grains, so they are subgrains. These observations show that the most grain boundaries in the samples deformed at the temperatures up to 400°C are low angle boundaries. However, it should
Table 3.2  The average subgrain size of 7075Al/SiCp composite at different strain rates and temperatures

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>$\dot{\varepsilon} = 1$ s$^{-1}$</th>
<th>$\dot{\varepsilon} = 0.1$ s$^{-1}$</th>
<th>$\dot{\varepsilon} = 0.01$ s$^{-1}$</th>
<th>$\dot{\varepsilon} = 0.001$ s$^{-1}$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Subgrain size (μm)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
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<td>*</td>
<td>*</td>
<td>1.19</td>
<td>1.56</td>
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<td>2.51</td>
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<tr>
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<td>2.5</td>
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<td>4.72</td>
</tr>
<tr>
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<td>2.46</td>
<td>3.77</td>
<td>5.12</td>
<td>5.8</td>
</tr>
</tbody>
</table>

Table 3.3  The average subgrain size of 7075Al alloy at different strain rates and temperatures

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>$\dot{\varepsilon} = 1$ s$^{-1}$</th>
<th>$\dot{\varepsilon} = 0.1$ s$^{-1}$</th>
<th>$\dot{\varepsilon} = 0.01$ s$^{-1}$</th>
<th>$\dot{\varepsilon} = 0.001$ s$^{-1}$</th>
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<tbody>
<tr>
<td></td>
<td>Subgrain size (μm)</td>
<td></td>
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</tr>
<tr>
<td>250</td>
<td>*</td>
<td>*</td>
<td>1.68</td>
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</tr>
<tr>
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<tr>
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<td>4.8</td>
</tr>
<tr>
<td>450</td>
<td>2.49</td>
<td>3.75</td>
<td>5.15</td>
<td>5.9</td>
</tr>
</tbody>
</table>
Figure 3.19 TEM micrograph of the composite deformed at 300°C, 1 s⁻¹, with diffraction pattern from several individual grains.
Figure 3.20  a) TEM micrograph of the composite deformed at 400°C, 1 s⁻¹  b) diffraction pattern from area containing several grains A, B, C, D and E  c) the diffraction pattern from the boundary between C and D. The same diffraction pattern with a zone axis of [111] indicates low misorientation between these grains.
be noted that a few large angle boundaries are found in these samples. But the size and the shape of the area enclosed by these highly misoriented grain boundaries indicate that they are previous grain boundaries and not the new recrystallized grains formed during deformation.

At 450°C, the deformed samples exhibit a few highly misoriented small grains at matrix/particle interface. Figure 3.21 depicts the presence of several small grains near a SiC particle with diffraction pattern and zone axis from individual grains in the sample deformed at 450°C, 1 s⁻¹. The grains A and B show a same zone axis of [T 22], but with different directions, the angle between these two directions is more than 25°. The grain C shows the same zone axis of [013], while the zone axis in grain D is [T 12]. The angle between the zone axis of two neighbour grains is considered as the angle or misorientation between the two grains (Usui et al., 1986c). It is found that the angle between the grains A and C as well as the angle between the grains C and D are also more than 25°. While in the matrix far away the SiC particles, a selected area consisting of several grains (Fig. 3.22) shows a same diffraction patterns suggesting they are subgrains with low misorientation between them.

The highly misoriented small grains near the particles indicate that these grains might be new dynamically recrystallized grains formed during deformation. These results indicate that local dynamic recrystallization may occur in the composite around the SiC particles during deformation at 450°C. However, it should be noted that the dynamic recrystallized grains were not replaced all the deformed samples and recovered substructure was dominant in the samples deformed at 450°C.
Figure 3.21 TEM micrograph of the composite deformed at 450°C, 1 s⁻¹ and diffraction pattern and zone axis from individual grains A, B, C and D. The different diffraction pattern and zone axis in these grains indicate that these are high misoriented grains.
Figure 3.22 TEM micrographs of the composite deformed at 450°C, 1 s⁻¹, in area far from the SiC particles. The diffraction pattern with a zone axis of $[\overline{1}12]$ from the area enclosed in circle indicates the low angle between these grains.
Chapter Three

3. 3 Discussion

3. 3. 1 Flow Behaviour at Elevated Temperatures

The shape of the flow curves of the reinforced alloy exhibits steady state value after initial strain hardening at low strains. These curves are of typical form in metals which show occurrence of dynamic recovery during deformation at high temperatures which is due to balance between generation and annihilation of dislocations (McQueen and Jonas, 1975; 1984). The similarity in the shape of flow curves in the composite and the monolithic alloy indicates that the similar restoration processes occur in these materials. The local dynamic recrystallization observed at 450°C does not influence the flow behaviour of the composite. This indicates that the presence of SiC particles in the matrix has no significant effect on deformation mechanism at high temperature. The reason for such phenomena is that the dislocations are able to climb around the large SiC particles so that deformation zones are not able to accumulate dislocations responsible for strain hardening (Humphreys et al., 1990; Sakaris and McQueen, 1992b). The interparticle spacing between SiC particles also is too large to accumulate dislocations around the particle to cause strain hardening at high temperature. The interparticle spacing between SiC particles in the present composite was found to be about 15 μm. The following formula was used to calculate interparticle spacing between SiC particles (Pandeys et al., 1992)

\[ \lambda = \frac{1}{2} d \left[ \sqrt{2\pi / 3V_f} - \sqrt{8/3} \right] \]

(3 - 5)

where \( \lambda \) is the average interparticle spacing between particles, \( d \) is the average particle size and \( V_f \) is the volume fraction of the particles.
The results show that strain rate sensitivity, m, values in both materials increase as temperature increases. The reinforced alloy shows a slightly higher m value compared to the unreinforced alloy in the temperature range used in this work. The possible reason for this may be due to a finer initial grain structure in the composite compared to the monolithic alloy. Mahoney and Ghosh (1987) have demonstrated that normal micrograin superplasticity can occur in aluminium MMCs containing 10% vol. SiC particles of 5 μm diameter, when these are processed to a very fine grain size by thermomechanical processing. However, the m values observed in the 7075Al/SiCp composite are in the range observed in aluminium based MMCs. For example, Chanda et al (1991) found m value between 0.15-0.2 in temperature range of 325-475°C in Al-Li SiCp composite.

The stress exponent, n, of the reinforced alloy is slightly higher than that of monolithic alloy. High n values have been found in SiC/Al composites during creep and deformation at high temperatures (Pickens et al., 1987; Park et al., 1990; Sakar and Surappa, 1993). The reason for the high n value in these composites produced by powder metallurgy was considered due to very fine oxide particles present in the aluminium matrix as a result of manufacturing which gave rise to the existence of threshold stress. This is because the analysis of the creep data by various threshold stress showed that the threshold stresses introduced by SiC particles were too small to account for the observed high n value in the composite (Park et al., 1990). The low n value in the present composite can be attributed to the presence of large SiC (average 14 μm) which are not able to act as effective barriers to dislocation movement at high temperatures and the absence of fine oxide aluminum in the present composite made by ingot metallurgy in comparison to the composites produced by powder metallurgy. Furthermore, theories based on dislocation climb during creep predict a low stress exponent of up to 4 (Nieh et al., 1988). The low stress exponent (< 4) in the composite and the matrix alloy obtained in this work also confirms that the dynamic recovery is principal mechanism at elevated temperatures.
The activation energy for deformation, \( Q \), is 168 kJmole\(^{-1} \) for the composite and 152 kJmole\(^{-1} \) for 7075Al alloy. The activation energy of the 7075Al alloy is similar to \( Q \) value reported by Evangelista (1991) in 7075Al alloy and to that for self-diffusion of aluminium (McQueen et al., 1991a). The composite shows a slightly higher activation energy than that the matrix alloy. The higher activation energy in SiC/Al composites compared to their matrix alloys has been reported during deformation or creep at elevated temperatures (Pickens et al., 1987; Sakaris and McQueen, 1992a; Alunni et al., 1993). High activation energy in the composites is attributed to the much higher flow stress in the composites compared to the matrix alloys at lower temperatures and similar flow stress in reinforced and unreinforced alloys at higher temperatures. It should be noted that the \( Q \) value is found to be higher compared to the monolithic alloy in this work, but it is far lower than those reported in 6061 or 2024 aluminium based composites (in the range of 280 to 380 kJmole \(^{-1} \)).

The possible reason for the lower activation energy in the present composite can be attributed to the effect of matrix alloy. This is because 7075 aluminium alloy is a high strength aluminium alloy so that the presence of SiC particles has a small effect on flow stress of the reinforced alloy as compared to the lower strength aluminium alloy. For example, the flow stress of the reinforced alloy is only 28\% higher than that the unreinforced alloy at 250°C, while the flow stress a 6061 aluminium composite is 50 to 30\% at 300 and 400 compared to the monolithic alloy (Sakaris and McQueen, 1992a).

The higher activation energy in the present composite compared to the monolithic alloy can be ascribed to the higher temperature dependence of flow stress in the reinforced alloy. The variation of flow stresses as a function of temperature at strain rates of 1 and 0.1 s\(^{-1} \) for the composite and the monolithic alloy is shown in Fig. 3.23. It can be seen that the flow stress decreases as temperature increases in both materials, however, the rate of decrease is faster in the composite compared to the matrix alloy. The composite
Figure 3.23 The flow stresses as a function of deformation temperature for the 7075Al/SiCp composite and 7075Al alloy at a) 1 s\(^{-1}\) b) 0.1 s\(^{-1}\) c
exhibits a higher flow stress at lower temperatures, but the difference in flow stress between two alloys decreases as temperature increases and these materials show almost similar stress levels at 450°C.

3.3.2 Effect of Temperature on Strength of the Composite

The 7075Al/SiCp composite shows a higher strengths (Yield stress and flow stress) under low temperature and high strain rate conditions of deformation in comparison with the monolithic alloy (Figs. 3.10 and 3.22). The effect of SiC particles on strength in the reinforced alloy significantly diminishes as temperature increases or as strain rate decreases and at 400 and 450°C at strain rates below 1 s⁻¹, both materials show almost similar strength. The composite also shows a higher strain rate hardening at low temperatures and high strain rates compared to the monolithic alloy, but both materials exhibit similar value at 450°C. These observations suggest that SiC particles can not act as dislocation barriers during deformation at high temperatures. This is because the SiC particles are too large and the interparticle spacing between SiC particles, ~15 µm in the present composite, also is large so that SiC particles can not act as effective barrier for dislocation at high temperature. The higher strength of the reinforced alloy compared to the unreinforced alloy at lower temperature can be attributed to the smaller substructures in the composite due to presence of SiC particles.

In the light of strengthening mechanism in MMCs, there are two important sources which contribute to strengthen of these materials. One of them is generation of dislocations due to the large difference between the coefficient of thermal expansion (ΔCTE) of aluminium and SiC (Arsenault and Shi, 1986). These dislocations are generated when the composites are cooled from the elevated temperatures of annealing or processing and present in the composites before deformation. This results in an enhanced internal stress.
and small subgrains. Another source for generation of dislocations in the composites is incompatibility between a deforming matrix and a non-deformable hard particle (Humphreys et al., 1990). This means that dislocations will be generated at the particles during deformation. This causes formation of unrelaxed dislocation structures such as Orowan loop at low strains and deformation zone at high strains.

During deformation at lower temperatures, the high dislocation density resulting from above sources restricts the plastic flow of the matrix and contribute to the strengthening and strain hardening. But as temperature increases the effect of mismatch between particle and matrix as well as ΔCTE on generation and accumulation of dislocations will change. This is because the dislocation cross slip and diffusion controlled climb increase with increasing temperature. As a result, the dislocation concentration at particles will be decreased with increasing deformation temperature. The interactions between particles and dislocations are also reduced when the temperature is increased and the strain rate is decreased (Liu et al., 1991). This indicates that the advantages in the strengthening by high dislocation density due to ΔCTE and geometrical mismatch between particle and matrix are reduced at high temperatures. These could be the reasons that strength and strain hardening of the reinforced alloy decrease more rapidly compared to the unreinforced alloy as observed in the present work.

Additionally, the another possible reason for the more decrease of the strength in the composite compared to the monolithic alloy at higher temperature may be due to local dynamic recrystallization observed in the composite.

The flow stress may be estimated from the dislocation density, from the cell size, or from the subgrain size in the MMCs in the appropriate deformation regime. In hot deformed composites, the subgrain size may usefully be taken as the strengthening parameter (Hasen and Barlow, 1993). The flow stress, \( \sigma \), shows a relatively linear relationship
Figure 3.24 The plot of flow stress, $\sigma$, as function of reciprocal of subgrain size in the 7075Al/SiCp composite.
with reciprocal subgrain size, \( d^{-1} \), in the composite, Fig. 3.24, according to the following equation:

\[
\sigma_c = 169.97 \, d^{-1} - 2.93
\]  

(3.6)

This indicates that the flow stress can be inversely related to subgrain size developed during deformation at elevated temperature (McQueen et al., 1991a). Thus, subgrain can be considered as a strengthening parameter in the composite. This linear relationship is in agreement with the results reported in aluminium alloys (Sellars, 1992; McQueen, 1977).

3.3.3 Microstructural Development in the Composite During Hot Deformation

The shape of flow curves indicates that dynamic recovery is the dominant restoration process during deformation at elevated temperature. The optical microstructures of the deformed composite show elongated grains after deformation at temperatures below 450°C. At 450°C, the microstructure contains several small equiaxed grains along with elongated grains having serration grain boundaries. The presence of equiaxed grains after deformation in aluminium alloys has been interpreted as a result of dynamic recrystallization (McQueen et al., 1991a). However, it could be due to the impingement of scalloped grains boundaries at large strains rather than formation of new grains (Humphreys, 1982). In the present work, the composite exhibits a few highly misoriented grains in the vicinity of particles and these grains could be considered as recrystallized grains formed during deformation. The possible reason for the occurrence of dynamic recrystallization in reinforced alloy seems to be due to high constrains in some areas between the large SiC particles. DRX in the aluminium based composites at high temperatures could be due to: firstly through particle enhanced nucleation (Tuler et al., 1988) and secondly due to strain gradient between the large ceramic particles (Xia et al. (1993b). However, it should be noted that the presence of high volume fraction of
recovered substructure with low misorientation between them above 400°C, as confirmed by SADP technique, indicates that dynamic recovery is the main restoration mechanism.

TEM observations show that the sample deformed at 250°C exhibits very fine cellular cell structure consisting of high dislocation density along with fine precipitates distributed in the matrix indicating low level of dynamic recovery at lower temperatures. But as temperature increases and strain rate decreases, the substructures become larger and the subboundaries become sharper. The subgrain morphology also changes from elongated to equiaxed as temperature increases. The pinning effect of precipitates also decreases with increasing temperature due to coarsening of precipitates and greater mobilities of dislocations (Evangelista et al., 1986). The large dislocation density produced during deformation at elevated temperatures can readily be annihilated and rearranged into ordered patterns and this is due to thermally activated assisted cross slip and climb of the dislocations into low energy configuration which results in the formation of equiaxed subgrains.

The subgrain size developed during deformation at high temperatures can be related to the temperature compensated strain rate (Z parameter). The variation of 1/d with ln Z is plotted in Fig. 3.25 for both the composite and the monolithic alloy. It can be seen that the subgrain size increases as Z parameter increases, i.e. temperature increases and strain rate decreases. Increasing the deformation temperature enhances thermal activation processes which make the dislocations more mobile. Decreasing the strain rate permits the greater number thermal activated events. Consequently, increasing of temperature and decreasing of strain rate result in more annihilation of dislocation, higher degree of dynamic recovery which leads to increase the subgrain size. The reciprocal subgrain size, \( d^{-1} \) shows an approximation linear relationship with log Z. The following linear equations can be written for the composite and the monolithic alloy:
Figure 3.25 The variation of subgrain size (1/d) as a function of ln Z for a) 7075Al/SiCp composite and b) 7075Al alloy
\[ d_{c}^{-1} = 0.16 \log Z - 1.114 \quad (7075\text{Al}/\text{SiC}) \quad (3-7) \]
\[ d_{m}^{-1} = 0.11 \log Z - 0.572 \quad (7075\text{Al}) \quad (3-8) \]

A comparison between two equations shows that the slope of the linear equation for the composite is higher than for the monolithic alloy which demonstrates that the coarsening of substructure in the reinforced alloy is faster than the unreinforced alloy as temperature increases and strain rate decreases. This can support the idea that the generation of dislocations due to ΔCTE and geometry mismatch between particle and matrix during deformation decreases and as a result of which the substructure will coarsen at a higher rate in the reinforced alloy compared to the unreinforced alloy.

It has been found that the presence of SiC particles in the matrix alloy results in non-uniform subgrain size in the composite. TEM micrographs show that the subgrains between the particles and near the particles are finer than the area far from the particles. This can be ascribed to the high dislocation density in vicinity of the SiC particles resulting from geometrical mismatch between SiC particle and matrix during deformation as well as dislocation generation due to ΔCTE.

The structural observations reveal a finer substructure in the reinforced alloy in comparison to the unreinforced alloy at low temperature and high strain rate, but a similar substructure is found in the both alloys at high temperature and low strain rate as can be seen from Table 3.1 and 3.2. The finer substructure in the composite compared to the monolithic alloy at lower temperatures can be attributed to higher dislocation density produced during deformation in the surrounding matrix and presence of prior high dislocation density due to ΔCTE. But as temperature increases, the effect of CTE and accumulation of dislocations around the particles decreases because the rate of cross slip
and climb of dislocations increases with increasing in temperature and the difference in subgrain size in the composite and the monolithic alloy decreases.

3.4 Conclusions

1. The 7075Al/SiCp composite exhibits higher flow stress than that the matrix alloy at lower temperatures and higher strain rates, but both materials show almost similar flow stress levels at 400 and 450°C at strain rates below 0.1 s⁻¹. The higher flow stress in the composite at lower temperatures is mainly due to the contribution of the dislocations and substructure induced by flow of matrix around the SiC particles. The drop in the flow stress in the composite at high temperature can be attributed to the higher diffusion rate and enhanced dynamic recovery.

2. TEM investigations and Selected diffraction pattern technique reveal the presence of substructure with low angle boundary between them. However, a few highly misoriented small grains are found in the samples deformed at temperatures above 400°C at the particle/matrix interface which could be considered as dynamically recrystallized grains. They nucleate in regions which are highly constraint between large particles.

3. The composite shows elongated subgrains with high dislocation density within the substructure at lower temperatures (below 300°C) which could be due to low rate of dynamic recovery at lower temperatures. At temperatures above 300°C, the subgrain morphology changes to equiaxed shape and also dislocation density decreases which implies that the recovery rate increases significantly as temperature increases and strain rate decreases.

4. The reciprocal subgrain size shows an approximation linear relationship with \( \log Z \) (Zener-Holloman Parameter). As temperature increases and strain rate decreases (high \( Z \) value), the subgrain size increases. The high slope in linear equation (0.16) for the
composite is indicative of strong strain rate and temperature dependence of subgrain size. The flow stress at high temperature also shows a relatively linear relationship with reciprocal subgrain size. This indicates that the subgrain size can be considered as a strengthening parameter in hot worked composites.

5. In comparison to matrix alloy, the composite material has smaller subgrain size and higher dislocation density as a result of lower dynamic recovery at lower temperatures and higher strain rates. But as temperature increases and strain rate decreases the difference in subgrain size between two materials decreases and at 450°C a similar subgrain size is observed in the reinforced and unreinforced alloy.

6. The plot of ln $\dot{\varepsilon}$ versus ln sinh ($\alpha\sigma$) are linear in the strain rate range tested with slopes fairly independent of temperature. The stress exponent, n, is found to be 3.28 and 2 for the reinforced and unreinforced alloy, respectively. The activation energy of the composite is found to be 168 kJ mole$^{-1}$ which is higher than that for the monolithic alloy (151 kJ mole$^{-1}$).
CHAPTER 4
Mechanical Behaviour During Double Pass Deformation and Static Recrystallization after Hot Working
4.1 Introduction

Metal forming of aluminium metal matrix composites is usually performed at elevated temperatures where the flow stress is relatively low and the ductility is high (Humphreys et al., 1990). In most hot working operations the overall shape is attained through a series of deformation steps or passes (McQueen and Jonas, 1985). Under such conditions, the metals are subjected to static restorations during interpass holding time at elevated temperatures (McQueen and Conrod, 1986). Although several studies have been undertaken on multi-stage deformation of aluminium and its alloys at high temperatures, as well as on hot workability of Al/SiC composites, limited information is available on multistage deformation behaviour and static restoration after hot working in discontinuous reinforced aluminium metal matrix composites. In this chapter, the effects of temperature and delay time between passes on the fractional softening are examined. Microstructural development during holding time and after double pass deformation is also studied. In addition, the static recrystallization after annealing the deformed samples at elevated temperatures has also been examined.

The double pass deformation was performed by compression testing at 300 and 400°C at a relatively high strain rate of 1 s⁻¹. All samples were deformed to a true strain of 0.4 in first pass and then held at the deformation temperature for different times (6 to 900 sec), and were compressed again to a further true strain of 0.5 followed by water quenching. The samples deformed in the temperature range of 250-400°C were annealed at or above deformation temperature (up to 500°C for 1 hour) to the study static recrystallization behaviour.
4. 2 Experimental Results

4. 2. 1 Flow Curves Characterization in Double Pass Deformation

Figure 4.1 shows the typical true stress true strain curves for 7075Al/SiC$_p$ composite after double-stage and single pass deformation at 300 and 400°C at a constant strain rate of 1 s$^{-1}$. It can be seen that at a given deformation temperature the flow curve after the first pass and subsequent second pass attains a steady state after initial rapid strain hardening. It can be seen that yield stress on reloading in second pass is lower compared to the flow stress just before unloading in first pass in these flow curves. The comparison of flow curves in double and single pass deformation shows that the continuous deformation flow curves slightly fall below the interrupted curves at both temperatures. Similar flow curves were obtained for the unreinforced alloy.

4. 2. 2 Fractional Softening in the Composite and in the Monolithic Alloy

Fractional softening is determined mechanically from the drop in yield stress during a holding interval in proportion to the strain hardening that occurred during the first pass using the following equation (McQueen and Jonas, 1975; 1985):

\[
\%FS = \frac{\sigma_2 - \sigma_3}{\sigma_2 - \sigma_1}
\]

Where $\sigma_2$ is the flow stress immediately before unloading in the first pass, $\sigma_3$ is the yield stress on restraining and $\sigma_1$ is the original yield stress as shown in Fig. 4. 2. The variation of fractional softening as a function of delay time for the composite at 300 and 400°C at a strain rate of 1 s$^{-1}$ is shown in Fig. 4.3. The $\%$ FS was found to be 16% after 6 sec of interpass hold time at 300°C and this value of fractional softening remained
Figure 4.1 The comparison of true stress true strain flow curve in single pass and double pass deformation for the 7075Al/SiCp composite at 300 and 400°C. The delay time between two passes was 300 sec.
Figure 4.2 Schematic flow stress for determination of fractional softening

Fractional Softening = \( \frac{\sigma_2 - \sigma_3}{\sigma_2 - \sigma_1} \)

\( \sigma_2 \) = Flow stress on unloading in the first pass

\( \sigma_3 \) = Yield stress on reloading (second pass)

\( \sigma_1 \) = Yield stress in the first pass
Chapter Four

Figure 4.3 The variation of fractional softening vs delay time for 7075Al/SiCp composite at 300 and 400°C
unchanged even when the hold time was increased to 60 sec. However, 25% FS was found when the delay time between passes increased to 600 sec and this remained the same even when the delay time was extended to 900 sec. For deformation temperature of 300°C, it can be seen that the total change in fractional softening is about 9% as the delay time increases from 6 to 900 sec. At 400°C, fractional softening varied from 22 to 43% as the delay time was raised from 6 to 900 sec.

Figure 4.4 shows the %FS vs delay time at 300 and 400°C for the monolithic alloy. At 300°C, %FS was 20% when a delay time of 6 sec was given in rest period between passes, and it increased to 26% when the delay time was increased to 900 sec. At 400°C, as the delay time was increased from 6 to 900 sec, the %FS varied from 25 to 48%. It can be seen from Figs 4.3 and 4.4 that for a given delay time, both materials show a higher fractional softening at 400°C compared to 300°C. For example, the FS increases from 16 to 26% in the composite and from 22 to 30% in the monolithic alloy as the deformation temperature is increased from 300 to 400°C with an interpass hold time of 60 sec between two passes.

Figure 4.5 compares %FS as a function of delay time for the reinforced and unreinforced alloys at 400°C. It can be seen that the effect of delay time on FS for the monolithic alloy is similar to that for the reinforced alloy. However, the amount of %FS in the unreinforced alloy is slightly higher than for the reinforced alloy which indicates that the presence of SiC particles has no significant effect on the fractional softening of the reinforced alloy.

4. 2. 3 Microstructural Development During Double Stage Deformation

Figure 4.6 shows the optical microstructure of the 7075Al/SiCp composite after double-stages deformation at 400°C with a delay time of 900 sec between the two passes. It can
Figure 4.4 The changes of fractional softening as a function of delay time for 7075Al alloy at 300 and 400°C
Figure 4.5 The changes of fractional softening as a function of delay time for the reinforced and unreinforced alloy at 400°C
be seen that the microstructure exhibits elongated grain structure with serrated grain boundaries in the direction normal to the compression axis. The serration of grain boundaries are believed to be result from grain boundary migration via interactions with intersecting sub-boundaries (Humphreys, 1982). Similar microstructural features were found in deformed samples with various delay times as well as the sample deformed continuously to a strain of 0.9. The microstructures of the samples deformed at 300°C with different rest times between the two passes were also quite similar to that of sample given single pass deformation. Similar features are observed in the unreinforced alloy as shown in Figure 4.7 which shows the microstructure of the monolithic alloy given double deformation at 400°C with a delay time of 900 sec between the two passes. The microstructural observations revealed elongated grains in the samples given single or double pass deformation at a given temperature.

Figure 4.8 displays TEM micrographs of the reinforced alloy after interrupted straining at 300°C with two different hold times of 60 and 900 sec. The presence of substructure with dislocations inside the subgrains as well as interaction of fine precipitates with dislocations and subboundaries can be seen in these samples. The subgrain size found to be similar suggesting that the final substructure is not sensitive to delay time between two passes.

TEM microstructures of the composite after double stage deformation at 400°C with delay times of 300 and 900 sec are shown in Fig. 4.9. A well-defined substructure can be seen in these micrographs. TEM micrographs of the unreinforced alloy (Fig. 4.10) after interrupted straining at 400°C with different delay times also show an equiaxed recovered substructure with sharp subboundaries.

The comparison of the structures deformed at 300 and 400°C shows that the substructure becomes equiaxed and the subboundaries become sharper as temperature varies from 300 to 400°C in both reinforced and unreinforced alloys. The composite exhibits a finer
Figure 4.6  Optical microstructure of the 7075Al/SiCp composite after double pass deformation at 400°C with a delay time of 900 sec.

Figure 4.7  Optical Microstructure of the 7075Al alloy after double pass deformation at 400°C at a strain rate of 1 s⁻¹ with a delay time of 900 seconds
Figure 4.8 TEM micrographs of the reinforced alloy after double pass deformation at 300°C, 1 s$^{-1}$ with different delay time a) 60 sec b) 900 sec
Figure 4.9  TEM micrographs of the composite deformed by double pass at 400°C, $1 \text{s}^{-1}$ with various delay times a) 300 sec b) 900 sec
Figure 4.10  TEM photographs of the monolithic 7075Al alloy after double pass deformation at 400°C with delay time of a) 300 sec b) 900 sec
substructure compared to the monolithic alloy at these temperatures irrespective of interpass hold time.

The microstructures of the composite compressed to a strain of 0.4 (first pass) at 300°C and the deformed sample held at this temperature for 900 sec are shown in Fig. 4.11a and b, respectively. A high dislocation density inside the substructure can be seen in both samples. Similar microstructural features are observed in the monolithic alloy as shown in Fig. 4.12. This indicates that dislocation density does not decrease significantly during holding time at 300°C.

Both reinforced and unreinforced alloys exhibit a recovered substructure when the samples are held for 900 sec at 400°C after the first pass deformation as shown in Fig. 4.13 and Fig. 4.14, respectively. But no static recrystallization was detected in these materials at 400°C. However, it was found that the subgrain size was slightly larger in the sample held for 900 sec compared with the sample quenched immediately after the first pass. These observations indicate that subgrain growth commences during holding time at 400°C in both reinforced and matrix materials.

The average subgrain size obtained in the reinforced and unreinforced alloys at different conditions are summarized in Tables 4.1 and 4.2. Several observations can be made from the data in these Tables. It can be seen that as delay time increases from 6 to 900 sec, the subgrain size after the second pass does not change significantly at 300°C or 400°C. The composite exhibits subgrain size of about 0.98 and 1.9 μm after double stage deformation at 300 and 400°C, respectively. On the other hand, the monolithic alloy has subgrain size of about 1.35 and 2.2 μm under similar deformation conditions. These results indicate that delay time has no significant effect on final substructure and the subgrain size remains unchanged during the hold interval at 300°C, but increases slightly at 400°C. For example, at 400°C the composite shows a subgrain size about 1.9 μm after the first pass,
**Figure 4.11** The TEM microstructure of the reinforced alloy deformed at 300°C a) after first pass b) held for 900 sec at 300°C after the first pass

**Figure 4.12** TEM micrographs of the matrix alloy deformed at 300°C a) quenched after the first pass b) held at 300°C for 900 sec before quenching after the first pass
Figure 4.13  TEM micrographs of the composite deformed at 400°C, 1 s⁻¹ after straining to a true strain of 0.4 and held at 400°C for 15 min before quenching.

Figure 4.14  TEM micrographs of the monolithic alloy deformed at 400°C, 1 s⁻¹ after straining to a true strain of 0.4 and held at 400°C for 15 min before quenching.
Table 4.1  The average subgrain size of the composite at different conditions at a strain rate of 1s\(^{-1}\)

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>strain</th>
<th>delay time (sec)</th>
<th>subgrain size (µm)</th>
<th>No. pass</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>0.9</td>
<td>60</td>
<td>0.95</td>
<td>2</td>
</tr>
<tr>
<td>300</td>
<td>0.9</td>
<td>600</td>
<td>0.97</td>
<td>2</td>
</tr>
<tr>
<td>300</td>
<td>0.9</td>
<td>900</td>
<td>0.98</td>
<td>2</td>
</tr>
<tr>
<td>300</td>
<td>0.9</td>
<td>0</td>
<td>0.95</td>
<td>2</td>
</tr>
<tr>
<td>300</td>
<td>0.4</td>
<td>0</td>
<td>0.93</td>
<td>2</td>
</tr>
<tr>
<td>300</td>
<td>0.4</td>
<td>900</td>
<td>0.93</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.9</td>
<td>60</td>
<td>1.87</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.9</td>
<td>300</td>
<td>1.9</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.9</td>
<td>600</td>
<td>1.94</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.9</td>
<td>900</td>
<td>1.95</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.4</td>
<td>0</td>
<td>1.85</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.4</td>
<td>900</td>
<td>1.9</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.4</td>
<td>900</td>
<td>2.1</td>
<td>2</td>
</tr>
</tbody>
</table>

Table 4.2  The average subgrain size of 7075Al at different conditions at a strain rate of 1s\(^{-1}\)

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>strain</th>
<th>delay time (sec)</th>
<th>subgrain size (µm)</th>
<th>No. pass</th>
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</thead>
<tbody>
<tr>
<td>300</td>
<td>0.9</td>
<td>6</td>
<td>1.3</td>
<td>2</td>
</tr>
<tr>
<td>300</td>
<td>0.9</td>
<td>900</td>
<td>1.3</td>
<td>2</td>
</tr>
<tr>
<td>300</td>
<td>0.9</td>
<td>0</td>
<td>1.3</td>
<td>2</td>
</tr>
<tr>
<td>300</td>
<td>0.4</td>
<td>0</td>
<td>1.28</td>
<td>2</td>
</tr>
<tr>
<td>300</td>
<td>0.4</td>
<td>900</td>
<td>1.28</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.9</td>
<td>300</td>
<td>2.2</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.9</td>
<td>600</td>
<td>2.35</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.9</td>
<td>900</td>
<td>2.4</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.9</td>
<td>0</td>
<td>2.16</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.4</td>
<td>0</td>
<td>2.2</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.4</td>
<td>600</td>
<td>2.45</td>
<td>2</td>
</tr>
<tr>
<td>400</td>
<td>0.4</td>
<td>900</td>
<td>2.56</td>
<td>2</td>
</tr>
</tbody>
</table>
and it increases to 2.1 μm when the sample is held for 900 sec. Under the same conditions, subgrain size in the monolithic alloy is about 2.2 μm after the first pass and increases to 2.56 μm as delay time increases to 900 sec. The samples deformed to strain of 0.4 exhibit similar subgrain size to those samples strained to 0.9 in both composite and matrix materials, whether single pass or double pass. These results suggest that the level of strain does not affect subgrain size indicating a stabilized substructure in these materials.

4.2.4 Static Recrystallization After Hot Working

Reinforced and unreinforced samples were deformed to a true strain of 0.9 in the temperature range of 250-400°C (at a strain rate of 1 s\(^{-1}\)) and annealed at the deformation and higher temperatures up to 500°C for 1 hour. TEM micrographs of the composite and the monolithic alloy deformed at 250°C and subsequently annealed at 350 and 400°C are shown in Figs. 4.15 and 4.16, respectively. It can be seen that the both materials show fully recrystallized structure at 400°C. At 350°C, the reinforced alloy exhibits recovered substructure with recrystallized nuclei around the SiC particle. In contrast the unreinforced alloy shows only recovered substructure. This indicates that the large SiC particles accelerate recrystallization in the reinforced alloy compared to the unreinforced alloy. Table 4.3 shows the effect of the annealing and deformation temperature on static recrystallization in the composite.
Figure 4.15  TEM micrographs of the composite deformed at 250°C, 1 s⁻¹ and then annealed for 1 hour at (a) 350°C (b) 400°C

Figure 4.16  TEM micrographs of 7075Al alloy deformed at 250°C, 1 s⁻¹ and subsequently annealed for 1 hour at (a) 350°C (b) 400°C
Table 4.3  Effect of deformation and annealing temperature on recrystallization in the 7075Al/SiCp composite

<table>
<thead>
<tr>
<th>Deformation temperature (°C)</th>
<th>Annealing temperature (°C)</th>
<th>250</th>
<th>300</th>
<th>350</th>
<th>400</th>
<th>450</th>
<th>500</th>
</tr>
</thead>
<tbody>
<tr>
<td>250</td>
<td>N</td>
<td>N</td>
<td>N*</td>
<td>R</td>
<td>R</td>
<td>R</td>
<td>N</td>
</tr>
<tr>
<td>300</td>
<td>N</td>
<td>N*</td>
<td>R</td>
<td>R</td>
<td>R</td>
<td>N</td>
<td>N*</td>
</tr>
<tr>
<td>350</td>
<td>N</td>
<td>N*</td>
<td>R</td>
<td>R</td>
<td>N</td>
<td>N*</td>
<td>R</td>
</tr>
<tr>
<td>400</td>
<td>N</td>
<td>N*</td>
<td>R</td>
<td>N</td>
<td>N*</td>
<td>R</td>
<td>N</td>
</tr>
</tbody>
</table>

N = No recrystallization
N* = few recrystallized nucleus at particles
R* = Not complete recrystallization
R = Complete recrystallization

This Table shows that no recrystallization takes place during annealing at deformation temperature. But complete recrystallization takes place when the samples are annealed at temperatures much higher than the deformation temperature. It can be seen that when the samples are deformed at lower temperatures (250 and 300°C) complete recrystallization is found at annealing temperatures which are at least 150°C above that of the deformation temperature. But in the samples deformed at 350 and 400°C, complete recrystallization is found when annealing temperature is 100°C higher than the deformation temperature.

Figure 4.17 shows the optical micrographs of the composite samples deformed in the temperature range of 250-400°C and then annealed at 500°C. It can be seen that the sample deformed at 250°C shows fine grain size after annealing, but as deformation temperature increases the grain size increases and a very large grain size is observed in the sample deformed at 400°C. A similar behaviour was found in the monolithic alloy. These observations indicate that at a given annealing temperature, grain size increases as deformation temperature increases in both reinforced and unreinforced alloys.

Tables 4.4 and 4.5 show the average recrystallized grain size in the deformed samples of the reinforced and unreinforced alloys, after annealing at different temperatures, respectively. The data from these Tables shows that in the samples deformed at 250°C,
Figure 4.17 Optical micrographs of the 7075Al/SiCp composite annealed at 500°C for 1 hour after deformation at a strain rate of 1 s⁻¹ at a) 250°C b) 300°C
Figure 4.17 Optical micrographs of the 7075Al/SiCp composite annealed at 500°C for 1 hour after deformation at a strain rate of 1 s⁻¹ at c) 350°C d) 400°C
**Table 4.4** Average recrystallized grain size in the hot deformed 7075Al/SiCp composite after annealing at different temperatures

<table>
<thead>
<tr>
<th>Annealing temperature (°C)</th>
<th>Grain size (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$T_d = 250°C$</td>
</tr>
<tr>
<td>400°C</td>
<td>33</td>
</tr>
<tr>
<td>450°C</td>
<td>23</td>
</tr>
<tr>
<td>500°C</td>
<td>25</td>
</tr>
</tbody>
</table>

**Table 4.5** Average recrystallized grain size in the hot deformed 7075Al alloy after annealing at different temperatures

<table>
<thead>
<tr>
<th>Annealing temperature (°C)</th>
<th>Grain size (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$T_d = 250°C$</td>
</tr>
<tr>
<td>400°C</td>
<td>45</td>
</tr>
<tr>
<td>450°C</td>
<td>34</td>
</tr>
<tr>
<td>500°C</td>
<td>53</td>
</tr>
</tbody>
</table>

$T_d =$ Deformation temperature
the grain size decrease as annealing temperature increases from 400°C to 450°C, but it increases as annealing temperature increases from 450 to 500°C in all samples deformed at other temperatures. It can be seen that the composite exhibits a finer grain size than that the monolithic alloy under the same conditions.

4. 2. 4. 1 Effect of Strain Rate on Recrystallization Behaviour in the Composite

The deformed samples in the range of temperature between 250-400°C at various strain rates (1, 10⁻¹ and 10⁻² s⁻¹) were annealed at 500°C for 1 hour. Figure 4.18 displays a group of optical micrographs of the composite taken from the samples deformed at 300°C at strain rates of 10⁻¹ and 10⁻² s⁻¹ after annealing. It can be seen that the grain size increases as strain rate decreases. A similar trend was observed in the samples deformed at 250 and 350°C after annealing. But the samples deformed at 400°C at strain rates of 10⁻¹ and 10⁻² s⁻¹ did not show recrystallization after annealing. Figure 4. 19 shows the optical and TEM micrographs of the composite deformed at 400°C, 10⁻¹ s⁻¹ and annealed at 500°C. This sample shows unrecrystallized grain structure containing substructure. The variation of recrystallized grain size in the composite after annealing at 500°C as a function of strain rate is shown in Figure 4.20. It can be seen that the grain size after annealing increases as strain rate decreases. In addition, at a given strain rate, the recrystallized grain size is finer in the samples deformed at lower temperatures in comparison to the samples deformed at higher temperatures.
Figure 4.18  Optical micrographs of the 7075Al/SiCp composite annealed at 500°C for 1 hour after deformation at 300°C at a) 0.1 s\(^{-1}\) b) 0.01 s\(^{-1}\)
Figure 4.19  The microstructures of the composite after annealing at 500°C for 1 hour after deformation at 400°C, 0.1 s⁻¹ a) optical micrograph b) TEM micrograph showing the presence of substructure
Figure 4.20 The changes of recrystallized grain size vs strain rate in the composite annealed at 500°C for 1 hour
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4. 3 Discussion

4. 3. 1 Flow Behaviour in Double Pass Deformation

The flow curves of the 7075Al/SiCp composite and the monolithic alloy show a steady state regime after initial strain hardening after the first as well as the second pass deformation. This implies that the high temperature mechanical behaviour after reloading in second stage is similar for the reinforced and unreinforced alloys and the fact that the large SiC particles do not seem to affect the flow behaviour of the material at high temperatures. The shape of the flow curves indicates that dynamic recovery occurs during deformation at high temperatures in both materials.

The yield stress on reloading is lower than the flow stress on unloading in the first pass and this can be attributed to the occurrence of static restoration during delay time. It is known that in multipass deformation at constant temperature and strain rate, the metal undergoes static restoration during interval, so that its yield stress is lower than that at the end of the previous pass (McQueen et al., 1984). Fractional softening which is generally determined from the drop in yield stress during interval depends on deformation temperature and extent of delay time. The effect of deformation temperature and holding time on FS in these materials is discussed in the following section.

4. 3. 2 Effect of Temperature and Delay Time on Fractional Softening

The results of the present work show that FS increases as temperature and delay time increase in both reinforced and unreinforced alloys and this can be attributed to the increased rate of static restoration processes with increasing temperature or time as restoration processes are thermally activated processes which depend on both temperature and time. The thermally activated processes are also diffusion controlled, so as the hold
time increases at a given temperature, more diffusion occurs and hence more static restoration takes place (Evans and Dunstan, 1971; McQueen and Jonas, 1985).

However, the composite and the monolithic alloy show a lower FS than that the aluminium and its alloys under similar deformation conditions. For example, a maximum FS are found in the composite and the matrix alloy to be 43 and 48% at 400°C with the delay time of 900 sec, respectively while under similar deformation conditions, the aluminium shows 50% FS after a short delay time of 20 sec (McQueen and Ryum, 1985).

The effect of delay time on FS is also marginally in these materials compared with aluminium and its alloys. Figure 4.21 compares the variation of fractional softening as a function of delay time for pure aluminium (Evans and Dunstan, 1971), Al-Mg% alloy (Sellars et al., 1986), the 7075Al/SiCp composite and 7075 aluminium monolithic alloy under similar conditions. It can be seen that the aluminium and Al-Mg alloy show a sharp increase in fractional softening as delay time increases, but the composite and the 7075 aluminium alloy show gradual increase in FS as delay time increases. The sharp increase in FS with delay time in aluminium and Al-Mg alloy is due to the occurrence of static recrystallization. The absence of a rapid increase in FS with delay time in the present reinforced and unreinforced 7075 aluminium alloy can thus be attributed to the absence of static recrystallization during hold time. The composite exhibits slightly lower FS compared to the monolithic alloy at all delay times. This can be attributed to the interaction of SiC particles with dislocations and subgrain boundaries which can affect the subgrain growth in the composite during holding time. It should be noted that subgrain growth during delay time in the reinforced alloy is less than in the unreinforced alloy (Tables 4.3 and 4.4). However, it seems that the large SiC particles have no significant effect on fractional softening of the reinforced alloy. This suggests that the important factor which affects the fractional softening in these alloys is the presence of fine precipitates and dispersions in the matrix alloy.
Figure 4.21 The variation of fractional softening as a function of delay time for pure aluminium (Evans and Dunstan, 1971), Al-Mg alloy (Sellars et al., 1986), and 7075Al/SiCp composite and 7075 aluminium alloy
It is worth mentioning that three kinds of second phases containing constituent, dispersoids and hardening precipitates exist in 7075Al matrix alloy. Coarse constituent phases are relatively large (2-5 μm) and consist of insoluble and or partially soluble intermetallic compounds. These compounds primarily form as a result of interaction of the alloying elements with impurity elements such as Si and Fe (Raghavan et al., 1985). Dispersoids which are also intermetallic compounds, are relatively smaller in size (0.5 to 2 μm) and typically contain elements such as Mg, Cu, Zn and Cr which are intentionally added for either strengthening or retardation of recrystallization kinetics. They are Al₈Mg₃Cr₂ compounds and form in both triangular and spherical morphologies. Hardening precipitates (< 0.01 μm) in size, are Zn rich, consisting of GP zones and transition phases with partially coherent interfaces. They are the (MgCu)Zn₂ phase (M phase or equilibrium precipitates) which form during the ageing treatment as an extremely fine and homogeneous dispersion and these precipitates are responsible for the high strength exhibited by the 7000 series alloy (Evangelista et al., 1988). EDS analysis of the precipitates on the present 7075 aluminium alloy after annealing indicates that the microstructure consists of coarse and fine precipitates of (MgCu)Zn₂ phase, which are same as the dispersoids and constituent phases reported by Evangelista et al. (1988; 1991). Among of these phases, dispersions and fine precipitates have the most significant effect on stabilization of substructure.

The effect of fine particles and dispersions on fractional softening in metals during multistage deformation has been reported (McQueen et al., 1984; 1989b). These authors found 28% of FS in Al-4.5Mg-0.8 Mn alloy which contained fine Al₆Mn particles deformed at 400°C at strain rate of 1 s⁻¹, and held for 20 sec after the deformation (McQueen and Ryum, 1985), while under similar conditions, the FS in pure aluminium was ~ 50% (Evans and Dunstan, 1971) suggesting definite influence of dispersions on
fractional softening. The low fractional softening in the present materials can also be
ascribed to the stabilizing effect of fine MgZn2 precipitates and dispersions in these
alloys.

4. 3. 3 Effect of precipitates on Microstructural Changes During Delay
Time

It is known that the fractional softening in multistage deformation is caused by static
restoration processes occurring in metals during interpass hold period and this has a
significant effect on the mechanical behaviour in the following deformation passes. Static
restorations taking place during hold time at elevated temperature include recovery and
recrystallization. This implies that the amount of FS can be related to the microstructural
changes such as subgrain growth and static recrystallization during hold time. However,
the presence of dispersions and fine precipitates can stabilize the substructure and retard
static recrystallization (McQueen and Jonas, 1985).

The low fractional softening observed in the present materials is evidenced by the minor
microstructural changes that accompany during holding time. Figure 4.22 shows the
pinning of dislocations and subboundaries with fine precipitates after the first pass at
300°C following holding for 900 sec at the same temperature. The interactions of
dislocations and subboundaries with precipitates can reduce the mobilization of
dislocations and subboundaries. It should be noted that the subgrain size does not change
significantly even after 900 sec of delay time between the two passes at 300°C, as can be
seen from the data in Tables 4.1 and 4.2. This indicates that the recovered substructure
after the first pass is so stable that somewhat small static recovery occurs during the
interval time. The flow stress on reloading in the second pass is also found to be slightly
higher than that after first pass, Fig. 4.1, although the yield stress is lower than the flow
stress on unloading because of static recovery during the hold interval. This behaviour
Figure 4.22 Interaction of dislocations with precipitates after first pass at 300°C and holding at deformation temperature before quenching a) 7075Al/SiC composite b) 7075Al alloy
might be attributed to structural changes occurring during holding time, possibly precipitation which lead to increase in the strength, or solution of large particles, which can give solute strengthening.

Marginal subgrain growth is observed during holding at 400°C in both reinforced and unreinforced alloys as can be seen in Tables 4.1 and 4.2. This is possibly due to increasing mobility of dislocations and the decreased pinning effect of fine precipitates as temperature increases from 300 to 400°C. In spite of this, microstructural observations do not show static recrystallization during interpass hold time at 400°C. Sheppard et al. (1986) have also observed subgrain growth in aluminium during holding time after hot rolling. Evans and Dunstan (1971) have observed a sharp decrease in dislocation density during holding time and some recrystallized grains with well defined boundaries within subgrain arrays after a short time in aluminium deformed at 410°C. In the present work, although dislocation density decreases and subgrain size slightly increases during the holding time at 400°C, but dislocation precipitation interactions are still observed in the samples even after holding for a long time. The subgrain growth observed during holding time is also much less than that in aluminium alloys. It is envisaged that the absence of static recrystallization might be due to limited subgrain growth in these alloys which suggests that fine precipitates and dispersions in these alloys stabilize the substructure and thus prevent sufficient subgrain growth to produce recrystallized grains.

4.3.4 Microstructural Development During Double Pass Deformation

The observed flow curves as well as microstructural examinations indicate that dynamic recovery takes place in the composite during deformation at high temperature either on interrupted or continuous straining. The selected area diffraction pattern (SADP) taken from the deformed composite after double pass deformation confirms that dynamic recovery is the dominant mechanism. The SADP from the composite sample given
double pass deformation with a delay time of 300 sec between two passes at 400°C is shown in Fig. 4.23. It can be seen that the selected area consisting of subgrains A, B, C, D, E and F shows only one set of diffraction patterns with a zone axis of [001]. These observations indicate that the most subboundaries have low misorientation in the composite deformed by either continuous or interrupted straining at high temperatures. A similar behaviour is found in samples deformed by single pass deformation as discussed in chapter 3.

Reinforced and unreinforced alloys show a recovered substructure after the first pass at both temperatures. In addition, the samples deformed to the strains of 0.4 (first pass) and 0.9 (second pass) do not show significant difference in subgrain size. This means that new substructure with a similar size forms again during second pass deformation from the prior substructure. Two mechanisms have been suggested for the reformation of new subgrains from the previous substructure during deformation (McQueen and Conrod, 1986). The first is the unravelling and reknitting of the subboundaries. In this model, the existing boundaries are unravelled completely before the new boundary can form. Another mechanism of sub-boundary formation in a prior substructure is the unpinning of dislocations followed by glide and by interactions with other dislocations to form new boundaries. In this mechanism, unpinning of dislocations occurs because of the action of a pile up on a weakly formed boundary and these unpinned dislocations may glide, cross slip and interact with other dislocations to form new boundaries.

TEM reveals similar recovered substructures in the samples deformed by interrupted or continuous testing at a given deformation temperature, regardless of the rest time between the two passes. This indicates that delay time between two passes has no significant effect on final substructure and that mainly deformation temperature determines the final substructure. It has been reported that the final equilibrium substructure is dependent on
Figure 4.23  a) TEM micrograph of the 7075Al/SiC composite after double pass deformation with a delay time of 300 sec at 400°C, 1 s⁻¹ b) diffraction pattern from selected area containing subgrains of A, B, C, D, E and F
deformation conditions such as temperature and strain rate (McQueen and Jonas, 1975). A similar behaviour has been reported in aluminium and its alloys during multistage deformation (Farag et al., 1968; McQueen et al., 1984). McQueen et al. (1984) found a similar substructure in Al-5Mg-0.8Mn alloy deformed by either multistage or single deformation at 300-450°C. It has also been found that multipass rolling does not have a significant effect on the microstructure of aluminium and that the deformation conditions in the final pass control the final substructure (Sheppard et al., 1986).

4.3.5 Effect of Deformation and Annealing Temperature on Static Recrystallization Behaviour After Hot Working

The results show that static recrystallization does not occur in deformed samples on annealing at the deformation temperature in both reinforced and unreinforced alloys. However, SRX has been observed when the annealing temperature is at least 100°C above the deformation temperature in these materials.

It has been reported that nucleation of recrystallized grain is inhibited by closely spaced particles because dislocation and sub-boundary motion is retarded thus inhibiting subgrain growth (Humphreys, 1977; Zaidi and Sheppard, 1983). Furthermore, Humphreys and Martin (1968) have pointed out that the presence of dispersoids stabilize slip and these lattice misorientation across the slip planes are reduced. Such stabilized dislocation configurations are highly resistant to annealing because of the absence of large local misorientations. It should be noted that the significant level of dynamic recovery can not provide sufficient strain energy to cause static recrystallization. The microstructural observations in TEM support the above explanation. Fig. 4.24 shows the microstructure of the composite strained at 1 s⁻¹ at 400°C and subsequently annealed at 450°C. A well recovered substructure with precipitates inside the subgrains and subboundaries can be seen clearly. The possible reasons for this behaviour in the present alloys seem to be due
Figure 4.24  TEM microstructure of the composite deformed at 400°C, 1 s⁻¹ and subsequently annealed at 450°C for 1 hour showing the presence of substructure with precipitates in subboundaries.
to the occurrence of dynamic recovery during deformation and the presence of dispersoids which stabilize the substructure. This is consistent with the results reported by Belling et al. (1990) on SRX in Al-1Mn-1.2Mg alloy after hot working.

4.3.5.1 Effect of Large SiC Particles on Recrystallization Behaviour of the Composite After Hot Working

The large SiC particles affect the starting recrystallization temperature especially in the samples deformed up to 350°C. The sample annealed at 350°C after deformation at 250°C shows few recrystallized nuclei in the vicinity of SiC particles but this is not the case in the unreinforced alloy under the same deformation conditions, Figs 4.15a and 4.16a. This suggests that nucleation of SRX grains occurs at the particles and large SiC particles could accelerate SRX during annealing after hot working.

It is known that recrystallization in composites occurs due to particle stimulated nucleation (PSN) during annealing after cold working (Humphreys, 1988; Liu et al., 1989). The presence of hard non-deformable particles during deformation induces strong deformation gradients which are associated with both microstructure refinement and lattice orientation gradients (Furu et al., 1993). During cold working, the dislocations generated at the interface between the particle and the deforming matrix are localized and this leads to the formation of deformation zones adjacent to large particles (Humphreys, 1977; Humphreys et al., 1990). In deformation zones at large particles the dislocations are arranged in small subgrains with a relatively large misorientation across the sub-boundaries (Liu et al., 1991). During annealing, recrystallization occurs in the deformation zones adjacent to the particles (Humphreys et al., 1990).

It has been further suggested that increasing the temperature of deformation may change the PSN activity. During annealing after cold working, recrystallization due to PSN is
restricted by the growth criteria of deformation zones, i.e. recrystallization would only be nucleated from deformation zones. But if deformation temperature increases, the situation may arise in which dislocations annihilate at the same rate as they arrive at the particles and thus no deformation zones will be formed (Furu et al., 1993). Humphreys (1988) has also suggested that PSN will only occur if deformation zones are formed at the particles, and therefore deformation temperature must be below the transition temperature. It seems that the conditions required for PSN to control recrystallization during annealing following hot working are defined by either formation of deformation zones or the growth criterion depending on deformation temperature. The results of the present work show that SiC particles seem to stimulate recrystallization when the composite is annealed after deformation at lower temperatures, i.e. formation of a deformation zone results in stimulation of recrystallization. At higher deformation temperature the growth of the deformation zone occurs and the particles can not stimulate the recrystallization.

PSN has a pronounced effect on recrystallized grain size in the annealed samples after deformation at low temperatures. The sample deformed at 250°C followed by annealing at 500°C shows a fine grain size, but a very large grain size is observed in the sample deformed at 400°C and annealed at 500°C. This indicates that large reinforcement particles restrict grain growth in the composite under certain conditions. In line with the above explanation, a high dislocation density can be generated around the particles at lower deformation temperatures and these can act as sites for nuclei and thus enhance nucleation. Such situation can result in fine recrystallized grains after annealing, thus PSN seems to be responsible for fine grain structure in the samples deformed at lower temperature but annealed at high temperature after deformation. As deformation temperature increases the dislocations generated during deformation can be removed from the particles by climb, and in such a situation, there would be no advantages for the large particles to stimulate nucleation of recrystallization during annealing and this could lead to coarser structure as observed in Figure 4. 17 d.
4. 3. 4. 2 Effect of Deformation and Annealing Temperature on Recrystallized Grain Size

It has been found in this study that the recrystallized grain size after annealing at a given temperature increases with increasing deformation temperature, Tables 4.4 and 4.5. This change in grain size can be explained by the rapid rate of dynamic recovery as deformation temperature increases as well as the enhancement of recrystallization process in deformed structure driven by stored energy (Cotterill and Mould, 1976).

Recrystallized grain size can be influenced by annealing temperature. Liu et al. (1989) and Humphreys (1990) have reported that recrystallized grain size can be influenced by annealing temperature in two ways: If higher the annealing temperature employed and this could increase the nucleation rate thus results in larger recrystallized grain size. The reason being is that the rate of low-angle boundary migration can increase relative to the high-angle boundary migration rate with increasing annealing temperature resulting in higher nucleation relative to the growth rate. On the other hand, increasing the annealing temperature can cause the fine precipitates to grow coarser and lose some of the grain pinning ability leading to a high growth rate. If the former effect is strong, then recrystallized grain size becomes finer as annealing temperature increases, but if the later effect increases the recrystallized grain size increases with increasing temperature. The results of the present work show that grain size decreases as annealing temperature increases from 400 to 450°C in both materials deformed at 250°C, but it increase as annealing temperature increases from 450 to 500°C in all deformed samples, Tables 4.4 and 4.5. The decreasing of grain size at 450°C could be due to increasing of nucleation rate, whereas the increasing of grain size at 500°C can be due to inability of coarse precipitates to pinning the grains resulting in increasing grain size.
4. 3. 5. 3 Effect of Strain Rate on Recrystallization Behaviour of the Composite After Hot Working

The results show that the recrystallized grain size increases as strain rate decreases after annealing. For example, the recrystallized grain size after annealing at 500°C in the composite deformed at 250°C increases from 25 to 37 μm as strain rate decreases from 1 to $10^{-2}$ s$^{-1}$. This seems to be due to a decrease in nucleation rate because of increasing rate of dynamic recovery occurring at low strain rate. The samples deformed at 400°C and strain rates below 1 s$^{-1}$ did not show recrystallization even after annealing at 500°C and a TEM micrograph of the composite under such deformation conditions showed recovered substructure (Fig. 4.19b). These observations suggest that the microstructure in the annealed sample consists of substructure and recrystallization occurs in samples deformed at a critical strain rate which is dependent on deformation temperature. The reason for this is that as strain rate decreases at a given deformation temperature, there will be more time available for annihilation of dislocations so that the deformed structure can be recovered faster. These observations suggest that the recovered substructure in the sample deformed at high temperatures and low strain rates remains so stable that not enough stored energy or driving force are produced to initiate recrystallization during annealing.
4. 4 Conclusions

1. The 7075Al/SiCp composite and monolithic alloy show similar flow during double pass deformation, but the composite exhibits higher flow stress compared to the monolithic alloy at high temperatures.

2. The fractional softening increases with increasing deformation temperature and increase in delay time between the two passes. The composite and the monolithic alloy show maximum FS to be 43 and 48%, respectively at 400°C under strain rate of 1 s\(^{-1}\), with a holding time of 900 sec between two passes. But this fractional softening is much less than that reported for the pure aluminium and Al-Mg alloys. The low fractional softening in these alloys can be attributed to the presence of dispersions and fine precipitates in the matrix which stabilize the substructure.

3. TEM microstructures show recovered substructures in the reinforced and unreinforced alloys after double pass deformation at 300 and 400°C. Similar microstructure is also found in the samples deformed by continuous and interrupted straining at a given temperature. It is found that the delay time between the two passes has no significant effect on final substructure and the deformation temperature mainly determines the equilibrium substructure.

4. The subgrains remain almost unchanged during holding time after the first pass in composite and monolithic alloy at 300°C. While, marginal subgrain growth is observed in these materials during rest time at 400°C. The dislocation density has been found to not decrease significantly during holding time at 300°C, but slight decrease observed at 400°C. The absence of subgrain growth at 300°C can be ascribed to the stabilized substructures pinned by dispersions and fine precipitates. However, as temperature increases to 400°C, the mobility of dislocations increases and in addition gives rise to a decrease in pinning effect which could lead to slightly subgrain growth.
5. Static recrystallization (SRX) has been not found in the samples annealed at deformation temperatures. This can be attributed to the occurrence of dynamic recovery combined with the stabilizing effect of fine precipitates and dispersions in the matrix alloy. However, complete recrystallization is observed in both materials when the annealing temperature is 100 to 150°C above the deformation temperature.

6. Fine grain size is observed in the samples deformed at lower temperature and higher strain rate followed by annealing at high temperatures. This is because at lower temperatures, high density of dislocations are produced at particles and they can act as nucleation sites. This means particle stimulated recrystallization has more effect on grain size of samples deformed samples at lower temperatures after annealing, but as temperature increases the dislocations generated during deformation can be removed by climb so that the large particles can not stimulate nucleation. This leads to larger recrystallized grain size in the samples deformed at higher temperatures followed by annealing.

7. The recrystallized grain size increases as deformation temperature increases or strain rate decreases when the samples annealed at a given temperature. Static recrystallization does not occur in the composite deformed at 400°C and strain rate of $10^{-1}$ and $10^{-2}$ s$^{-1}$, but well recovered substructure is present in the composite samples after annealing at 500°C after deformation. This suggests that static recrystallization can occur at a critical strain rate depends on deformation temperature.
CHAPTER 5

Effect of Temperature and Heat Treatment on Tensile Properties and Fracture Behaviour of 7075Al/SiCp Composite
5.1 Introduction

The presence of brittle reinforcements in particulate reinforced MMCs, tends to reduce their ductility and fracture toughness. The current state of understanding of the relationship between microstructure and fracture behaviour of the composites is still not very clear. While there has been considerable research undertaken in evaluating the effects of reinforcement size and volume fraction on the fracture properties of aluminium based composites, limited studies have been undertaken on the effect of matrix microstructure on fracture behaviour of aluminium based MMCs. There has also been extensive study of steady state creep processes in discontinuously reinforced composites, but there is relatively little information in literature regarding tensile properties and failure mechanisms at elevated temperature. In this chapter, the effects of temperature and heat treatment on tensile properties and fracture behaviour of a 7075 aluminium alloy reinforced with 15% vol. of SiC particles are examined.

5.2 Results

Tensile tests were carried out on specimens in T6 and annealed conditions. The measurements of 0.2% proof yield stress, ultimate tensile strength (UTS), and tensile elongation were made from engineering strain-engineering stress curves. Optical and scanning electron microscopy were employed to study the fracture surface and longitudinal sections after fracture to determine the mode of fracture and the damage mechanism. TEM was also used to examine the details of microstructure of the composite samples.
5. 2. 1 Engineering-stress Engineering-strain Curves

The engineering-stress/strain curves of the 7075Al/SiC particulate composite at various temperatures (25-400°C) at a strain rate of $10^{-3}$ s$^{-1}$ in annealed and T6 conditions are shown in Fig. 5.1. It can be seen that at room temperature the composite exhibits maximum flow stress and the sample has little necking prior to failure. The tensile ductility is much lower in T6 condition compared to that in annealed state. As deformation temperature increases, the stress decreases and ductility increases. At 300°C, the composite attains peak and then with further straining the stress decreases steadily until fracture occurs. It should be noted that stress decreases more rapidly in the T6 sample compared to the annealed one. However, at temperatures above 300°C, the composite reaches peak stress at low strain and then reaches steady state level at higher strains in both T6 and annealed conditions. For example, at 350 and 400°C, there is hardly any difference in stress levels in two conditions.

Figure 5.2 compares the engineering stress/strain curves of the reinforced and unreinforced alloys in the temperature range from 25 to 400°C in annealed condition. The reinforced alloy shows a higher flow stress than the monolithic alloy at lower temperatures, while both materials exhibit almost similar flow stress at 350 and 400°C. It can be seen that the monolithic alloy shows a higher tensile ductility compared to the composite at all temperatures.

5. 2. 2 Tensile Properties at Room Temperature

Table 5.1 shows the tensile properties of the 7075Al/SiC particulate composite and the monolithic alloy at room temperature in T6 and annealed conditions. It can be seen that the reinforced and unreinforced alloys show much higher tensile strengths and lower ductility in T6 condition compared to annealed condition. The unreinforced alloy exhibits
Figure 5.1 Engineering-stress engineering-strain curves of the 7075Al/SiCp particulate composite at different temperatures at a strain rate of $10^{-3}$ s$^{-1}$ in T6 and annealed conditions.
Figure 5.2 Engineering stress - engineering strain curves of the reinforced and unreinforced 7075Al alloy at various temperatures at a strain rate of $10^{-3}$ s$^{-1}$ in annealed condition.
a higher yield stress and tensile strength than that in the reinforced alloy in T6 condition, whereas, in annealed condition, the reinforced alloy shows slightly higher tensile strength than that the unreinforced alloy. This indicates that the introduction of SiC particles in 7075 aluminium alloy decreases the tensile strengths of the reinforced alloy when the matrix attains peak strength. In annealed condition, however, SiC particles slightly increase the tensile strength of the reinforced alloy, but it decreases the tensile ductility in both conditions.

Table 5.1 Tensile properties of the composite and the monolithic alloy at room temperature in T6 and annealed (O) conditions

<table>
<thead>
<tr>
<th>Material</th>
<th>Yield Stress (MPa)</th>
<th>UTS (MPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7075Al/SiC-T6</td>
<td>480</td>
<td>540</td>
<td>5-6</td>
</tr>
<tr>
<td>7075Al/SiC-O</td>
<td>195</td>
<td>280</td>
<td>10-12</td>
</tr>
<tr>
<td>7075Al-T6</td>
<td>510</td>
<td>590</td>
<td>10-15</td>
</tr>
<tr>
<td>7075Al-O</td>
<td>190</td>
<td>275</td>
<td>23</td>
</tr>
</tbody>
</table>

5.2.3 Tensile Properties at High Temperatures

The mechanical strength of 7075Al/SiCp composite as a function of the temperature in annealed and T6 states is shown in Fig. 5.3. It can be seen that the yield stress and tensile strength decreases as temperature increases in both conditions. The composite in T6 temper exhibits a much higher yield stress and UTS at 250°C compared to the sample in annealed temper. As temperature increases the difference in the tensile strength in samples in two conditions decreases and finally in the temperature range from 350 to 400°C, the composite shows a similar strength in both conditions. These results suggest
Figure 5.3 Tensile strengths of the 7075Al/SiC_p composite as a function of temperature in annealed and T6 conditions
that complete annealing occurs in T6 samples during pre-heating and straining at these higher temperatures. A similar behaviour was shown by the unreinforced alloy.

The variation in average tensile elongation with temperature is shown in Fig. 5.4 for the composite in T6 and O temper (annealed) conditions. It can be seen that the tensile ductility increases as temperature increases up to 350°C and does not change significantly at 400°C in either conditions. The annealed samples show a higher tensile elongation compared to T6 samples up to 300°C but nearly similar elongation values are found above 300°C in samples in both T6 and annealed conditions. This suggests that the ductility is not sensitive to heat treatment at temperatures above 300°C. It should, however, be noted that the composite also exhibited similar tensile strength in two conditions at temperatures above 300°C.

The mechanical strengths (UTS and 0.2% YS) of the reinforced and unreinforced alloys as a function of temperature in annealed condition are shown in Fig. 5.5. It can be seen that the mechanical strengths follow a similar trend, i.e. the strength decreases as temperature increases for both alloys. The composite is stronger than that the monolithic alloy at lower temperatures, up to 300°C, but both materials display similar strengths at higher temperatures, above 300°C. This indicates that the reinforced alloy shows slight improvement in tensile strength properties at lower temperatures, but not at temperatures above 300°C compared to the monolithic alloy. This confirms that SiC particles have an insignificant contribution to strength of the composite at elevated temperatures. It can be seen from Fig. 5.6 that the tensile elongation of the composite is less than that of the monolithic alloy at all temperatures. The ductility in the matrix alloy increases as temperature increases, while the composite shows a maximum elongation at 350°C and it remains nearly constant as temperature is raised to 400°C.
Figure 5.4 Variation of tensile ductility of 7075Al/SiCp composite with temperature in annealed and T6 conditions
Figure 5.5 Mechanical strengths of the reinforced and unreinforced 7075Al alloy as a function of temperature in annealed condition
Figure 5.6  Tensile elongation vs temperature for the composite and the monolithic alloy in annealed condition
Tensile tests were carried out on both materials in annealed condition at three different strain rates at 400°C in order to compare the strain rate dependence of tensile ductility in the composite and the monolithic alloy at high temperature. The results are given in Table 5.2. It can be seen that the composite and the matrix alloy exhibit maximum tensile elongation at a moderate strain rate of $10^{-2} \text{ s}^{-1}$, although the reinforced alloy shows much lower tensile ductility compared to the unreinforced alloy under the same conditions. The maximum elongation was found to be 64% for the composite and 160% for the monolithic alloy at strain rate of $10^{-2}$ at 400°C.

### Table 5.2 Tensile elongation (%) for the composite and the monolithic alloy at 400°C at different strain rates

<table>
<thead>
<tr>
<th>Strain rate ($\text{s}^{-1}$)</th>
<th>7075Al/SiCp</th>
<th>7075Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>$10^{-3}$</td>
<td>45</td>
<td>118</td>
</tr>
<tr>
<td>$10^{-2}$</td>
<td>64</td>
<td>160</td>
</tr>
<tr>
<td>$10^{-1}$</td>
<td>56</td>
<td>135</td>
</tr>
</tbody>
</table>

5. 2. 4 Fracture of the Composite at Room Temperature

The fractured tensile specimens of the 7075Al/SiCp composite showed low ductility on the macroscopic scale in both annealed and T6 temper conditions, but the fracture surface revealed micro-void coalescence indicating ductile fracture mechanism. The typical fracture surface of the 7075Al/SiC particulate composite in annealed condition at room temperature is shown in the scanning electron micrograph in Fig. 5.7. It can be seen the equiaxed dimple appearance on fracture surface characteristic of ductile fracture of matrix. Figure 5.8 shows the fracture surface of the composite in T6 condition in which predominantly fractured particles surrounded by ductile fracture of the matrix can be observed. It is also seen that the number or frequency of particle fracture on fracture
Figure 5.7  Typical fracture surface of the 7075Al/SiCp composite after tensile test at room temperature in annealed condition

Figure 5.8  Fracture surface of the composite in T6 condition at room temperature
surface in T6 sample is more than that in annealed sample. The image analysis from micrographs of fracture surface in the composite revealed a considerable difference in volume fraction of fractured particle on fracture surface in two conditions. An area coverage of fractured SiC particles of 28% was observed in T6 material, whereas the annealed material exhibited only 5% of the surface covered by fractured particles.

It can be seen from Fig. 5.7 and 5.8 that there are two kinds of voids observable in the fracture surface of the 7075Al/SiCp composite. The first type of void nucleates either at debonding interfaces or at fractured particles and these are referred to primary voids and the dimensions of these voids were found to be between 4 and 15 μm. It should be noted that voids are found to be associated with SiC particles. The second type of void is nucleated in the matrix ligaments between the particles and these voids are known as secondary voids. The size of these secondary voids is about 0.6 μm which is much smaller compared to primary voids.

Optical micrographs of longitudinal section near the fracture surface of the tensile composite sample tested in annealed and T6 conditions are shown in Fig. 5.9. The damage in regions of high local segregation of particles can be seen with a few cracked particles behind the fracture tip in the annealed specimen. On the other hand, specimen in T6 condition shows extensive fractured particles which are aligned in the tensile direction and these are present in the region immediately below the fracture surface. It can also be seen that the particle fracture is more prevalent in T6 material compared to the annealed case. The fracture plane of most particles is perpendicular to the tensile loading suggesting that the particles are fractured by tensile stress induced in the particle. These observations indicate that cluster regions of particles are sensitive to premature damage in addition to the large particles which are more prone to fracture.
Figure 5.9  Longitudinal section of fractured tensile sample of the composite at room temperature a) annealed condition b) T6 condition
More details of fracture behaviour was examined by matching of both sides of the failed tensile samples and EDS (energy dispersive spectroscopy) X ray analysis of surface of SiC particles in fracture surfaces. The typical matching fracture fractographs from tensile specimen of the composite in T6 condition are shown in Fig. 5.10. Matching fractographs from both fracture surfaces shows that the majority of the particles in the fracture surfaces are cracked, i.e. remnants of particles can be matched perfectly on both halves of the fractured test pieces. The fractured particles can be seen in a, b, c, d and e in both sides of fracture surfaces. EDS analysis of the exposed surfaces of most of the particles in the fracture surface showed predominant Si peaks in T6 sample, Fig. 5.11. This suggests that there is no significant coating of aluminium on SiC particles, and particle fracture is the dominant fracture mode in T6 condition.

Matching fracture surfaces of the composite in the annealed temper are shown in Fig. 5.12. Evidence of decohesion between the SiC particles and the matrix can be seen in the regions of well-defined dimpled fracture at the SiC/matrix interface indicated by arrows in the SEM photographs of the composite strained to failure at room temperature. EDS analysis of the exposed particles on both matching surfaces reveals predominantly Al peaks as can be seen in Fig. 5.13. It seems that the decohesion occurs in the matrix leaving a thin coating of matrix on the SiC particles where the void formation would have initiated. These observations suggest that interface debonding is the main fracture mode in the annealed condition which is in sharp contrast to that for the T6 sample fracture mode at room temperature. The results show that particle fracture is the dominant fracture mode in T6 temper while in annealed condition, interface decohesion is the dominant mode. These results further show a significant effect of heat treatment on the fracture micromechanisms in 7075Al/SiCp composite at room temperature.
Figure 5.10 Matching fracture surface of the composite in T6 condition at room temperature. Particle fracture can be seen in a, b, c, d and e.
Figure 5.11  EDS analysis of the particles of a, b, c, d, e on fracture surface in Figure 5.10 showing Si peak
Figure 5.12 Typical matching fracture surface of the composite in annealed condition at room temperature. Interface decohesion can be seen in arrowed area.
Figure 5.13  EDS analysis of the particle (marked) on fracture surface in figure 5.12 showing Al peak
5. 2. 5 Fracture of the Composite at High Temperatures

All tensile specimens showed some necking before fracture at high temperatures. The SEM fractographs of the 7075Al/SiC particulate composite strained to fracture in the temperature range of 250 to 400°C in annealed condition are shown in Fig. 5.14 (a-d). It can be seen that all specimens show dimple appearance with plastic deformation of the matrix and the extent of plastic deformation of the matrix increases with increasing temperature. A similar fracture surface is observed in T6 condition at high temperatures as shown in Fig. 5.15. However, at 250°C, the specimen deformed in annealed condition shows a more plastic deformation of the matrix compared to T6 sample indicating a higher ductility in annealed specimen.

The close examination of the fracture surface at temperatures above 250°C exhibited the presence of precipitates on the SiC particles and inside the dimples in both conditions. Figure 5.16 shows the presence of precipitates on the SiC particles in the annealed sample failed at 400°C. EDS analysis revealed that these precipitates contained Mg, Zn, and Cu peaks which form equilibrium precipitate, (Mg,Cu) Zn₂. This was not the case in the specimen which failed at 250°C and also the most of dimples were associated with SiC particles. These observations indicate that at higher temperatures, void nucleation takes place in large precipitates in addition to matrix/particle interface, while at lower temperatures, the fracture is governed through the matrix as well as the matrix/particle interface.

The optical micrograph of the longitudinal cross section of the composite sample fractured at 400°C is displayed in Fig. 5.17 which shows internal damage associated with cluster of particles behind the fracture tip. This indicates that particle clustering regions are also preferred sites for void nucleation and damage at high temperatures.
Figure 5.14 Fracture surface of the composite in annealed conditions a) 250°C b) 300°C
Figure 5.14 Fracture surface of the composite in annealed conditions c) 350°C
d) 400°C
Figure 5.15  Typical fracture surface of the 7075Al/SiCp in T6 condition

(a) 250°C  
(b) 400°C
Figure 5.16 Fracture surface of the composite at 400°C in annealed condition showing the presence of precipitates on the surface of SiC particle

Figure 5.17 Optical micrograph of the longitudinal section of the composite at 400°C showing internal damage associated with cluster of particles behind the fracture surface
Figure 5. 18 is the SEM photomicrographs of longitudinal sections of tensile samples failed at 250 and 400°C in T6 condition. The details in the structure illustrate the nature of the damage immediately beneath tensile fracture surface. At 250°C, small voids can be seen at the particle ends, with little linkage between voids. But at 400°C, large voids at the ends of particles and in the matrix between the particles are seen. It is clear that the extent of growth and linkage of voids is extensive at 400°C compared to 250°C which suggests that the internal damage increases with temperature. Similar features are found in samples in annealed condition, Fig. 5. 19. However, at 250°C, the annealed composite shows more interparticle voiding and larger voids in the areas near the fracture surface in comparison to T6 material. Occasional particle fracture is found in regions of particle clustering and in large particles behind the fracture surface at high temperatures. The evidence of linking up of interface debonded was also observed at temperatures above 300°C. The comparison of fracture surface of the composite in T6 and annealed conditions do not show significant difference in fracture behaviour at high temperatures.

It should be noted that the fracture behaviour of the composite and unreinforced alloy at high temperature was quite different although both materials exhibited similar strengths at 350 and 400°C. The monolithic alloy exhibited more pronounced necking compared to the reinforced alloy and the ductility of the composite was also much lower than the matrix alloy at all temperatures. The unreinforced alloy exhibited localized plastic deformation during tensile test to a very small area at fracture at high temperatures. Figure 5. 20 shows the scanning electron micrographs of fracture surface and longitudinal cross section of the monolithic alloy at 400°C. It can be seen that the fracture surface near the fracture tip exhibits a few cavities indicating the final fracture occurs by a process of ductile rupture fracture.
Figure 5. 18  Backscattered scanning electron micrographs of the longitudinal section of the composite in T6 condition a) 250°C b) 400°C
Figure 5.19 SEM micrographs of the longitudinal section of the composite in annealed condition a) 250°C b) 400°C
Figure 5.20 Fracture surface (a) and longitudinal section (b) of the unreinforced alloy after fracture at 400°C showing little cavitation
The details of fracture examination in the 7075Al/SiC particulate composite at room and elevated temperatures reveal several features. Firstly, the matrix exhibits a significant effect on fracture behaviour at room temperature depending on structure in which the fracture mechanism shifts from particle cracking to interface failure as the microstructure changes from T6 to annealed condition. Secondly, in T6 condition, as temperature increases from ambient to high temperature the fracture mode changes from particle fracture to interparticle voiding. Thirdly, heat treatment conditions do not seem to show any significant effect on fracture behaviour at high temperatures, i.e. a similar fracture mode is observed in the composite both T6 and annealed conditions. Finally, it is found that at lower temperatures, the fracture is governed mainly by matrix and reinforcement, while at temperature above 300°C the linking of debonded particle/matrix interface is dominant.

5. 3 Discussion

5. 3. 1 Tensile Properties at Room Temperature

Several approaches have been taken in the literature to model the strength of MMCs such as modified shear lag theory. The modified shear lag model has been developed to predict strength of fiber reinforced composites. This model essentially involves the load transfer from the matrix to the fiber by means of shear stresses at the matrix-fiber interface. Nardone and Prewo (1986) have proposed the following formula to predict the yield strength of a particulate composite:

$$\sigma_{cy} = \sigma_{my} \left[ V_f \left( \frac{a + 4}{4} \right) / 4 + V_m \right]$$  \hspace{1cm} (5.1)

where $V_f$ and $V_m$ are the volume fraction of the particulate and matrix respectively and $a$ is the aspect ratio of the particle. Above equation precludes that the strengthening ratio
should only depend on volume fraction and aspect ratio, and not on the matrix properties. In addition, this equation anticipates a strength ratio, $\sigma_{cy}/\sigma_{my}$, larger than 1, i.e. higher yield stress in the composite compared to the monolithic alloy. The strength ratio in the present work was less than 1 in T6 and actually 1 in annealed condition. This is not consistent with the strength ratio more than 1 in the composites based on lower strength aluminium alloys. This indicates that the matrix type seems to play an important role in strengthening the composite at room temperature.

Humphreys (1988) has suggested that the addition of a brittle reinforcement in high strength aluminium alloys may even decrease the yield stress and strength of the reinforced alloy. The results of the present work also showed that the yield stress and UTS of the 7075Al reinforced with SiC particulate were less than that of the unreinforced alloy. Recently, Manoharan and Lewandowski (1992) have also observed a decrease in yield stress and UTS in 7091Al/SiC particulate composite in underaged and overaged conditions. However, it should be noted that in the composites based on lower strength alloys such as 6061, 1100Al alloys show an increased strength compared to their matrix alloys at ambient temperature.

Two possibilities have been suggested to explain the lower yield and tensile strength in composites based on high strength aluminium alloys compared to the monolithic alloy. It has been suggested that as the composites with high strength alloys matrices are strained, the stress on the reinforcement becomes large. Eventually fracture can take place in the brittle reinforcement in the presence of a pre-existing flaw, probably initiated during earlier processing. Once the reinforcement fractures the net load-carrying of the composite decreases and thus the strength may decrease (Humphreys, 1988; Manoharan and Lewandowski, 1992). The other possibility is that during deformation the mismatch between reinforcement and the matrix leads to a large stress concentration near the reinforcement and so the matrix may fail prematurely in that region (Levy and Papazian,
1990). But in lower strength matrices, the stresses reached locally might not be large enough for either of these effects to occur, resulting in improvement in strength at room temperature as reported in other works (Humphreys, 1988). The results of this work show that particle fracture is the main damage mechanism in T6 condition. This suggests that the first possibility outlined above could be the reason for the lower strength observed in the reinforced alloy compared to the unreinforced alloy.

The composite shows a lower tensile ductility compared to the unreinforced alloy at room temperature in both conditions, Table 5.1. It can be attributed to the higher potential for void nucleation and damage in the composite due to presence of SiC particles. The composite in T6 condition shows a lower tensile ductility compared with annealed specimen at room temperature. This indicates also that the behaviour of the matrix plays an important role in the determination of composite ductility.

5. 3. 2 Effect of Temperature on Tensile Properties

The experiments show that the composite exhibits a higher yield stress and tensile strength in compared with the monolithic alloy at lower temperatures up to 300°C, but this is not the case at temperatures above 300°C (Fig. 5. 3). The improvement in tensile strength in aluminium based composites at lower temperatures has been reported by Nieh et al.(1988a; 1988b) and Philips (1978). It has been demonstrated that the increased strength in particle reinforced composites over the unreinforced alloy is as a result of a much higher work hardening rate at lower strains (McDanelts, 1985) which is due to the constraint exerted by the elastic reinforcing particles on the plastic flow of the matrix: The reinforcing particles can only deform elastically, so that a much higher local matrix strain is required to accommodate any given plastic deformation in the composite than in the unreinforced alloy resulting in increased matrix stresses. However, this constraint can be relaxed by dynamic recovery as the strain increases and also as thermal recovery occurs as
the temperature rises (Zhao et al., 1994). It is expected that any misfit strain gradient should be quickly relaxed by recovery processes and this could lead to decrease in work hardening rapidly in temperature range used in this work. This is confirmed by tensile behaviour of the composite at temperatures above 250°C which shows that the work hardening rate is almost zero at very low strains.

The tensile strength decreases with increasing testing temperature in both reinforced and unreinforced alloys indicating a similar temperature dependence of tensile strength in both materials. This demonstrates that the microstructure of the matrix mainly dominates the strength of the composite and SiC reinforcements are too coarse to act as effective barriers to dislocation motion at high temperatures. Similar results have been found in other composites based on precipitation hardening aluminium alloys (Nieh et al., 1988b; Zhao et al., 1994).

The results show that the tensile ductility of the composite increases as temperature increases, but the composite does not exhibit strong temperature dependence of tensile ductility at temperatures above 300°C, while in the unreinforced alloy the ductility increases with increasing of temperature up to 400°C (Fig. 5. 6). The possible reason of this behaviour is that the composite has non-uniform distribution of SiC particles. It is considered that the increase in ductility with temperature in the reinforced alloy can also be due to greater ductility of the matrix alloy apart from decrease in local stresses around the hard SiC particles as a result of climb of dislocations around these particles (Goods and Brown, 1979). Such a situation could lead to fracture of the composite at higher strain at elevated temperature.

The present composite shows much higher tensile elongation compared with 9021Al/15%SiCp and 2014Al/20% SiCp composite at higher temperatures (Nieh et al. 1988b; Corbin and Wilkinson, 1989). The higher ductility in 7075Al/SiCp composite can be achieved from the easier flow of the matrix due to lower volume percentages of SiC
particles compared to that found in 2014/20%SiCp composite. The higher ductility in this composite in comparison with 9021Al/15%SiCp composite can be attributed to the absence of very fine Al$_2$O$_3$ and Al$_4$C particles in the 7075Al/SiCp composite. It is known that the dispersions on the grain boundaries can act as nucleation sites for cavities which lead to loss in ductility at high temperatures (Hawk et al., 1988).

It should be noted that the tensile elongation of the reinforced alloy is much lower than the unreinforced alloy at any particular temperature. The lower hot ductility in aluminium based composites has been reported by other authors (McQueen et al., 1994; Zhao et al., 1994). The major reason for this large difference in ductility is considered to be due to the presence of SiC particles that in turn lead to increased stress concentration at particles. High stress concentration would result in higher fissure nucleation and thus increased level of damage in the composite leading to fracture at low strains.

5.3.4 Effect of Heat Treatment on Tensile Properties

Both reinforced and unreinforced alloys exhibit a higher strength in T6 condition in comparison with the annealed case at lower temperatures. This indicates that the solute and precipitates influence the strengthening effect in these materials. It is known that during artificial ageing of Al-Zn-Mg alloys, the elements in supersaturated solution begin to precipitate, and the strength of the material increases as the number and size of the precipitates increase and eventually the strength reaches a maximum value (the peak aged condition) in T6 condition. The microstructure contains zones having an average diameter of 2 to 3.5 nm along with some transition precipitate, M', which is supposed to be the precursor of the equilibrium MgZn$_2$ (Hatch, J. E., 1984).

The increase in flow stress of composites in heat-treatable matrix is probably attributable to the additive effects of dislocation interaction with both the alloy precipitates and the
hard SiC reinforcement. This combination increases the lattice strain in the matrix, causing greater dislocation tangling which requires higher flow stresses to continue deformation (McDanels, 1985). But as temperature increases the effect of fine precipitates on strength diminishes due to precipitation coarsening resulting in a similar strength values in both T6 and annealed conditions.

5. 3. 5 Strain Rate Dependence of Tensile Elongation

Reinforced and unreinforced alloys exhibit a maximum elongation at an intermediate strain rate of $10^{-2}$ s$^{-1}$ at 400°C, Table 5.2. The strain rate influences of the ductility at high temperatures in several ways. Increasing of strain rate increases the stress concentration at grain boundaries, encouraging triple junction cracking, the incident of particle cracking and/or decohesion (Evangelista et al., 1986). On the other hand, the void growth increases as strain rate decreases at elevated temperatures (Kassner et al. 1991). This suggests that matrix softening and great tendency of cavitation due to decreasing strain rate have dual counteracting effects on ductility of metals at high temperatures. The matrix softening has a favourite effect on ductility, while void growth has a adverse effect on ductility. The enhancement of matrix softening as strain rate decreases is due to more time available for the thermally activated processes such as climb and cross slips whilst the increase in void growth rate with lowering strain rate could be due to higher diffusion.

The results of Pickens et al. (1987) showed that the hot ductility in 7090Al/SiC composite in torsion increased as strain rate increased from $10^{-3}$ to $10^{-1}$ s$^{-1}$ but decreased with further increase in strain rate at 427°C. However, Chanda et al. (1991) did not find a strong correlation between tensile ductility and strain rate in Al-Li alloy reinforced with SiC particles between strain rates of $10^{-5}$ and $10^{-1}$ s$^{-1}$ at 525°C but maximum elongation was attained at $5\times10^{-3}$ s$^{-1}$. The ductility in the present study shows a maximum at
$10^{-2}$ s$^{-1}$, but it decreases marginally at strain rate of $10^{-1}$ s$^{-1}$. The low ductility at very low strain rate seems to be due to increasing void growth, but such decrease in ductility with increasing strain rate could be due to the higher stress concentrations occurring around the particles (McQueen et al., 1994). The fracture surface of the sample strained at $10^{-2}$ s$^{-1}$ exhibited more plastic deformation of matrix compared to that deformed at $10^{-3}$ s$^{-1}$, although both samples showed dimple appearance on fracture surface. These observations indicate that the contribution of the matrix deformation in 7075Al/SiCp composite becomes increasingly where deformation rate is increased. A similar strain rate dependence of tensile ductility in the monolithic alloy supports the rate of matrix deformation on the ductility of the reinforced alloy.

5.3.6 Final Fracture Characteristics

The macroscopic observation of the tensile samples after fracture at room temperature shows brittle mode because of low ductility exhibited by composite, especially in T6 condition. But, the details SEM examination of fracture surface in the 7075Al/SiCp composite reveals wide range of dimples indicating ductile fracture of matrix. The presence of two types of voids (primary and secondary) on the fracture surface suggests that the primary voids are associated with SiC particles, and particles are found inside the large voids, Figs. 5.7 and 5.8. These voids could nucleate by the interface debonding or as a result of cracked particles (Ge and Schmauder, 1995). In the present composite, the possible source of formation of these voids in annealed material is found to be by interface debonding, Fig. 5.12, but in T6 condition it seems to be due to particle fracture, Fig. 5.10. The triaxial stress constraint around the particles could make the matrix harder than it would be in the absence of particles. The fine voids occur in the spacing between particles during straining and subsequently three stages of nucleation, growth and coalescence of voids then follow. However, these voids are found to be very small
and the plastic strain associated with void formation is quite small. As a result, the probabilities of void nucleation, growth and coalescence of these small voids are not high to impose tensile plastic features in the material. The low ductility of the composite is thus mainly determined by large voids and characterizes the brittleness property of the composite while the small voids have little influence on the ductile behaviour of the composite at room temperature.

Many studies on fracture behaviour of discontinuously reinforced metal matrix composites (DRMMCs) have shown that microvoid coalescence (MVC) is the predominate fracture mode (Hunt Jr. et al., 1987; Lewandowski and Liu, 1988; Whitehouse et al., 1991; Berdy, 1993). The void nucleation takes place at a particle/matrix interface by either cracking of the particles or decohesion of interface and then grow under the applied stress until a coalescence mechanism is activated, followed by total failure of the sample leading to a dimpled appearance.

The extension of these simple models developed to describe the ductile fracture of the materials containing low fraction of second phase to DRMMCs imply that the onset of void nucleation is the dominant factor. For example, according to Brown and Embury (1973), the growth strain becomes zero as volume fraction of particles increases to about 16% with assuming a uniform particle size and distribution. This indicates that the nucleation process dominates in materials with high volume fractions of reinforcements in DRMMCs. The absence of void growth in regions near the fracture surface at room temperature strongly suggests that void nucleation is dominant factor in fracture of the present material, Fig. 5. 9.

The longitudinal sections of the fractured composite show particle fracture in regions of cluster of particles and large particles at room temperature. Particle fracture depends on the local stress field on the particle exceeding the particle strength. Since the SiC reinforcement is brittle, thus the particle fracture strength is controlled mainly by the
intrinsic flaws. It has been found that larger particles are more prone to cracking because they have a higher probability of containing critical flaws.

The increased propensity for particle fracture in cluster of particles can be ascribed to the existence of high local stresses in these regions (Brown and Embury, 1973; Hunt Jr. et al., 1993). It is interesting to note that Flower and Watt (1988) have used finite element analysis to evaluate the effect of particle clustering on local stresses in the matrix in 6061Al/SiCp composite. Their results show an interparticle spacing of 0.736 times the particle diameter for a uniform distribution of particles and a particle spacing of 0.279 of the particle diameter for particles clustered along the loading direction. The decreased spacing results in about fifty percent increase in the local stresses. This is consistent with the many experimental studies including the present work showing that damage initiates in particle clusters in aluminium based MMCs.

The results of this work reveal a transition in fracture mode from particle fracture in T6 condition to decohesion and interface failure between matrix/particle in annealed condition at room temperature. This indicates a significant effect of matrix microstructure on fracture of the composite at room temperature. Two dominant void nucleation modes of particle cracking and decohesion at the particle/matrix interfaces have been observed in DRMMCs. It has been found that the void nucleation mode is sensitive to several microstructural parameters such as size, volume fraction of reinforcement, aspect ratio, matrix heat treatment and strain rate (Berdy, 1993).

It has also been reported a shift in fracture mode from particle cracking to decohesion of matrix in 7090Al/SiCp composite as temper condition changed from underaged (UA) to overaged (OA) by Lewandowski et al.(1988). It was suggested that the presence of precipitates in matrix/particle interface was the main reason for the fracture by decohesion and interface debonding. This was because the interfacial precipitates decreased the total interface strength so that these areas became favoured at certain favourable sites during
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tensile in overaged condition. TEM microstructure of the 7075Al/SiCp composite in both annealed and T6 conditions is shown in Fig 5. 21. The presence of large precipitates at the interface of the matrix and the particle as well as in the matrix in annealed condition is seen, while in T6 condition, this is not observed. A similar feature of microstructure is observed in annealed 7075Al/SiCp composite as well as in the overaged 7090Al/SiCp composite. But the precipitates are coarser in annealed 7075Al/SiCp composite in comparison with the overaged 7090Al/SiCp composite.

Although the presence of precipitates may decrease the interface strength and can be a reason for interface debonding in annealed condition, the difference in fracture behaviour in annealed and T6 conditions can not be explained only by the presence or the absence of interfacial precipitates in annealed and T6 condition as suggested by Lewandowski and Liu (1989). This is because the tensile strength of the composite in T6 condition is much higher than that in annealed condition, while in 7090/SiCp composite a similar tensile strength levels have been reported in UA and OA conditions. A possible alternative explanation for the discrepancy in fracture behaviour in T6 and annealed conditions could be a different level of stress concentration around the particles generated during straining. In T6 condition, the matrix strength is too high because of the presence of very fine precipitates and as a result, high stress concentration, sufficient to fracture particles, can be produced in the early stage of deformation around the particles. It is known that the fracture of particles depends on stress concentration at the particles which depends on the strength of the matrix as well as on the particle size (Humphreys et al., 1990). The much higher volume fraction of fractured particles (28%) in the fracture surface and in area below the fracture surface (Fig. 5. 9) in T6 material compared to the annealed one (6%) also supports the different stress level around the particles in two these conditions. In
Figure 5.21 TEM photographs of the 7075Al/SiCp composite in a) annealed condition exhibiting interfacial precipitates b) T6 condition
annealed state, the stress around the particles cannot reach to the sufficient level for particle to fracture and this is due to coarse precipitates and soft matrix which might lead to fracture by interface decohesion of the composite. The fracture observations show that the composite exhibits dimpled appearance indicating ductile fracture at high temperatures. Similar results have been reported in other studies in the composites based on aluminium alloys during creep, tensile or torsion tests at elevated temperatures (Pickens et al., 1987; Almas and Humphreys, 1988; Corbin and Wilkinson, 1989; Ma et al., 1993). It is considered that voids nucleate at the particle/matrix interface because the particle/matrix interface is a preferential site for cavity initiation due to presence of high stress concentrations around the particle during deformation.

At high temperatures, the examination of longitudinal sections of fractured composite shows the presence of cavities at matrix/reinforcement interface or at particles ends in the direction of the applied stress as well as in the regions where the SiC particles are clustered. The internal damage associated with cluster of particles and void size increases as temperature increases. Particle cluster regions and individual particle ends are two regions susceptible to void nucleation. It has been established that the regions of particle clustering exert a adverse influence on the resistance to damage initiation, and provide favourable paths for linkage of the damage (Hunt Jr. et al., 1993). This is because the degree of plastic constraint within the clusters is much higher than in the rest of the matrix. Shorter interparticle distance associated with such particle clustering would also provide the increased probability of linkage between neighbouring voids as a result of decreased propagation distances between initiation sites. This results in void nucleation in these regions of microstructure at lower levels of deformation and such areas also observed in the present composite.

Individual particle ends are regions of high matrix strain because the adjacent matrix is essentially free of constraint from adjacent particles. It is known that cavitation occurs in
a region of the matrix where the local stress has reached some critical value. It has been shown by continuum models that void nucleation takes place at the whisker ends and particle ends in uniformly distributed reinforcements. Nutt and Needleman (1987) have assumed in their analysis that void nucleation occurs when the normal stress to the interface is at a maximum.

The testing temperature influences the cavitation in metals containing second phase particles. At high temperatures, when dislocation mobility by climb is high, plastic relaxation is easy and local work-hardening is small, the rate of attaining the local stress could be decreased by allowing thermal or dynamic recovery. Under these conditions, nucleation of cavities is restricted (Goods and Brown, 1979). The examination of the longitudinal sections of the strained sample on the verge of fracture at 400°C did not exhibit void growth, while the fractured sample showed internal damage and large void in the matrix between the particles in addition at particles ends, Fig 5. 18. It should be mentioned that the SEM examination showed some voids around the SiC particles, but it was not clear whether that they nucleated during straining or were pre-existed in the composite and this was because the as-received composite also showed presence of voids at SiC particles, Fig. 5.22. These observations strongly suggest that void nucleation delays at high temperature, however, once cavitation begins, the extent or void formation increases rapidly with further straining. This indicates that the presence of large void in the area near the fracture surface is due to void growth. The increased internal damage and larger voids in the longitudinal sections of the fractured samples at 400°C in comparison to 250°C, can thus be attributed to the higher void growth at 400°C. Whitehouse and Clyne (1994) have also shown, by the method of measuring the sample density periodically during the tensile test, that the increasing temperature delays the void nucleation in Al/Al₂O₃ composite.
Figure 5.22  SEM photograph of as-received 7075Al/SiCp composite showing voids at particle end and particle fracture
The longitudinal section of the samples fractured at 250°C shows little debonding, while the samples tested at 400°C exhibit more debonding in areas below the fracture surface. The small extent of particle/matrix debonding at 250°C can be ascribed to strong bonding between particle and matrix so that the fracture path is through the matrix and reinforcement. But, at elevated temperatures a reduction in flow stress results in a substantial plastic flow even around the particles giving rise to enhanced debonding (Ghosh and Ray, 1987).

The results show that in T6 condition, particle fracture is the main damage mechanism prior to final fracture at room temperature, whereas interparticle voiding is the dominant mechanism at high temperatures. This is consistent with the results observed in 2014/SiCp and 2014Al/Al₂O₃ particulate composite (Corbin and Wilkinson, 1989; Zhao et al., 1994). Particle fracture requires the achievement of high local stresses within the reinforcement particle and this can only be attained when the matrix strength is high.

At room temperature, very high stress concentration can develop around the particle that is sufficient to fracture the particle. But at high temperature the local stresses are not high enough to fracture the particles, since the matrix flow stress decreases with increasing temperature, and in this case all the strain has to be accommodated by the matrix and eventually fracture occurs by void nucleation at the matrix/particle interface.

The presence of occasional fractured particles in longitudinal sections below the fracture surface in the samples tested at high temperatures seems to be more likely caused during manufacturing processing instead by tensile deformation at high temperatures. This is because the stress concentration around the particle at elevated temperature is too small to cause particles to fracture.
5.4 Conclusions

1. The reinforced alloy shows lower yield and tensile strengths compared to the unreinforced alloy at room temperature in T6 condition. This can be attributed to the fracture of particles at early stages of deformation due to high stress concentration around the particles leading to loss of load-carrying of the composite.

2. The composite exhibits a higher tensile strength when compared to the monolithic alloy at lower temperatures up to 300°C, but the similar strength levels observed at 350 and 400°C. This indicates that matrix microstructure dominates the strength of the composite at high temperature.

3. The composite shows a much lower tensile elongation than that the monolithic alloy over the whole temperature testing range which seems to be due to higher level of damage due to presence of SiC particles.

4. The fracture behaviour is significantly affected by heat treatment in the composite at room temperature. Particle fracture is the fracture mechanism in T6 condition, while interface decohesion is the dominant fracture mode in annealed condition. This can be ascribed to the different levels of stress concentrations around the particles in T6 and annealed conditions.

5. Particle fracture is the main damage mechanism prior to final fracture at room temperature, while interparticle voiding is dominant at high temperatures in the composite in T6 condition. In annealed condition, a similar fracture mode is observed at room and high temperatures, but the amount of plastic deformation of the matrix and void growth at high temperatures are much higher than that at room temperature.

6. A similar fracture behaviour is found in both T6 and annealed tempers at high temperatures. The composite exhibits a dimpled fracture surface indicating of ductile
fracture of matrix. The interval damage is found in the cluster of particles behind the fracture surface and the amount of damage increases as temperature increases.

7. The regions of clustered particles and large particles are found to be two prone sites for the failure by fracture of particles and void nucleation in the composite. At room temperature the particle fracture is observed at large particles and cluster of particles, whereas at high temperatures void nucleation is found in matrix between closely adjacent particles and particle ends.
CHAPTER 6
General Conclusions and Suggestion for Future Work
6.1 General Conclusions

In the present chapter, the main conclusions from the previous sections will be summarized. Suggestions will be then made for future work. The findings of present work has applicability in understanding the deformation behaviour of particulate reinforced 7075 aluminium alloy at elevated temperature from both mechanical behaviour and microstructural development aspects.

1. The true stress true strain curves of the composite obey the normal high temperature behaviour of the monolithic alloy. The flow stress increases rapidly, attains maximum level at low strain and then reaches a steady state at higher strain. The shape of flow curves is typical for the materials undergoing dynamic recovery during deformation at elevated temperature.

2. The reinforced alloy exhibits higher strength than that the unreinforced alloy at temperatures below 400°C, but both materials show similar strength levels at 450°C. The higher flow stress exhibited by the composite compared to the matrix alloy can be attributed to the higher dislocation density generated in the matrix due to presence of SiC particles during straining. The decrease in the flow stress in the composite at higher temperature is mainly due to the higher diffusion rate and enhanced dynamic recovery.

3. The activation energy, $Q$, for the composite is found to be 168 kJmol$^{-1}$, whilst for the monolithic alloy it is 151 kJmol$^{-1}$. The higher activation energy of the composite compared to the matrix alloy is indicative of the higher temperature dependence of flow stress in the former material compared to the monolithic alloy.

4. The microstructural studies reveal the presence of substructure in the composite at all temperatures and strain rates used in the present work. The selected area diffraction pattern (SADP) technique confirms that dynamic recovery is the dominant deformation
mechanism at high temperature. However, a few highly misoriented grains are observed around the SiC particles at temperatures above 400°C and it seems that some dynamic recrystallized grains form during deformation.

5. The composite exhibits elongated substructure with high dislocation density within the subgrains at lower temperature below 300°C. But as temperature increases the subgrain morphology changes from elongated to equiaxed and dislocation density decreases which implies that the rate of recovery increases significantly as temperature increases. In comparison to the matrix alloy, the composite material shows finer subgrain size and higher dislocation density at lower temperature and higher strain rate. But as temperature increases and strain rate decreases, the difference in subgrain size between the reinforced and unreinforced alloy decreases.

6. The composite and matrix alloy exhibit similar flow behaviour during double pass deformation. These materials show higher fractional softening (FS) at 400°C as compared to 300°C for a given delay time. The FS also increases marginally as holding time between two passes increases at a given deformation temperature. However, the FS values observed in these materials are less than those reported in aluminium and its alloys. The microstructural observations suggest that mainly static recovery occurs during holding time in these materials. This can be attributed to the presence of dispersion and fine precipitates in these materials.

7. Reinforced and unreinforced alloys showed no evidence of recrystallization after annealing between deformation temperatures from 250-400°C, but complete static recrystallization was observed when the samples were annealed at temperature 100 to 150°C above these temperatures. The absence of recrystallization after annealing can be ascribed to the dynamic recovery in the hot deformed samples combined with the stabilizing effect of fine precipitates, \((\text{MgZn})_2\) and other dispersions in the matrix.
8. The samples deformed at lower temperatures show fine recrystallized grains, whereas coarse grain structure is found in the samples deformed at higher temperatures followed by annealing. At lower temperatures, SiC particles can stimulate nucleation for recrystallization, but at higher deformation temperature, large particles can not stimulate nucleation resulting in coarse grains. The possible reason is that the dislocations generated during deformation at higher temperature can be removed by climb around the particles.

9. The unreinforced 7075 aluminium alloy exhibits a higher yield and tensile strength compared to the reinforced alloy in T6 condition at room temperature. The lower tensile strength in the composite in T6 condition can be attributed to the fracture of particles in early stage of deformation at very low strain, due to high stress concentration around the particles sufficient to fracture the particles leading to the loss of load-carrying abilities of the composite.

10. The fracture behaviour is significantly influenced by the matrix microstructure in the composite at room temperature. Particle fracture is dominant fracture mechanism in T6 condition, while interface debonding seems to be the principal fracture mechanism in annealed state. The difference in fracture behaviour in T6 and annealed conditions could be due to different level of stress concentrations around the hard particles.

11. Deformation temperature has a pronounced effect on the fracture behaviour of the composite in T6 condition. At room temperature, particle fracture plays an important role for overall damage prior to final fracture, but at high temperature interparticle voiding is dominant. Large particles and the regions of clustered particles are found to be prone sites for damage in the composite. At room temperature the particle fracture is found at cluster of particles and large particles regions, whereas at high temperature void nucleation is observed in matrix closely adjacent particles and particles ends.
6.3 Suggestions for Future Work

1. The effect of temperature and strain rate was studied on mechanical behaviour and microstructural development of 7075 aluminium alloy and 7075Al/SiCp in annealed condition (fully precipitated), but it would be important to examine how the presence of solute elements in solution can affect the workability of reinforced alloy.

2. In the present work, the effect of temperature and delay time on fractional softening was studied, but the effect of strain and strain rate would also be important. The future work should involve study of the effect of strain rate and strain on fractional softening of these materials as well as more detailed examination of microstructural changes occurring during hold time between multipass. It will be also interesting to examine the effect of strain and hold time on the recrystallized structure of the composite at high temperatures.

3. The effect of strain rate on ductility was studied only at 400°C. An investigation should be made on the effect of other heat treatments such as solution treated, underaged and overaged conditions as well as the examination of the effect of strain rate on fracture behaviour of the composite at low and high temperatures. In the present work, most of the samples strained to fracture and only few samples strained to a strain close to fracture strain at 400°C. The void nucleation and growth was observed in the longitudinal sections of the fractured samples, but it was not clear whether these voids were nucleated during tensile loading or they already existed in the as-received materials and then grew during tensile test at high temperature. It is, therefore suggested that future study should concentrate on detail of fracture mechanism from interrupted tests and examination of void nucleation using study by in-situ scanning electron microscopy.
Appendix

1. Calculation of $\alpha$ values for the 7075Al/SiCp composite and 7075Al alloy

In order to plot the data according to equation 3.2, the value of $\alpha$ must be known. As first approximation, $\alpha$ is taken as $\beta/n_1$. $\beta$ is obtained from following equation:

$$\dot{\varepsilon} = A_1 \exp(\beta \sigma)$$

at high stresses, where it is generally independent of temperature; $n_1$ is given by

$$\dot{\varepsilon} = A_2 \sigma^{n_1}$$

at low stresses, where it is considered as a temperature-independent constant. $A_1$ and $A_2$ are constants. The value of $\beta$ evaluated from plot of $\ln \dot{\varepsilon}$ vs $\sigma$ was found in a range from 0.079 to 0.13 over the temperature range 250 to 450°C for 7075Al/SiCp composite and from 0.106 to 0.13 for the 7075Al alloy. The $n_1$ values were calculated from the slope of $\ln \dot{\varepsilon}$ vs $\ln \sigma$, and the values was found decreased from 11.2 to 4.2 as the temperature increased from 250 to 450°C and from 13.5 to 4.75 for the monolithic alloy at the same conditions. For each $\beta$ it was found five $\alpha$ values, with different $n_1$ values. The $\alpha$ values obtained were used to calculate $n$ value, stress exponent, for each temperature

$$n = \left[ \frac{\delta \ln \dot{\varepsilon}}{\delta \ln \sinh (\alpha \sigma)} \right]_T$$

(3)

The most suitable value of $\alpha$ was the one which led to the minimum standard deviation ($S_d$) in an average $n$ over the full temperature range studied. It was found
that the best $\alpha$ value to fit the data according to the above equation and to obtain the minimum $S_d$ to be 0.027 for the composite and 0.058 for the monolithic alloy. The corresponding values of stress exponent $n$ were found to be 3.28 for the composite and 2.02 for the monolithic alloy. Tables 1A and 2A show the $\ln [\sinh(\alpha \sigma)]$ and $n$ value for the 7075Al/SiCp composite and 7075 aluminium monolithic alloy, respectively.

Table 1A calculation of $\ln [\sinh (\alpha \sigma)]$, $\alpha = 0.027$ for the 7075Al/SiCp composite

<table>
<thead>
<tr>
<th>strain rate(s$^{-1}$)</th>
<th>250°C</th>
<th>300°C</th>
<th>350°C</th>
<th>400°C</th>
<th>450°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4.3</td>
<td>3.35</td>
<td>2.68</td>
<td>1.84</td>
<td>1.03</td>
</tr>
<tr>
<td>0.1</td>
<td>4.05</td>
<td>2.81</td>
<td>1.73</td>
<td>1.03</td>
<td>0.036</td>
</tr>
<tr>
<td>0.01</td>
<td>2.95</td>
<td>2</td>
<td>1.03</td>
<td>0.4</td>
<td>-0.27</td>
</tr>
<tr>
<td>0.001</td>
<td>2.05</td>
<td>1.33</td>
<td>0.58</td>
<td>0.01</td>
<td>-1.02</td>
</tr>
<tr>
<td>n</td>
<td>2.92</td>
<td>3.31</td>
<td>3.21</td>
<td>3.65</td>
<td>3.31</td>
</tr>
</tbody>
</table>

$$S_d = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (n_i - \bar{n})^2}, \ N = 5 \ i = 1 - 5 \ \bar{n} = 3.28 \ S_d = 0.236$$

Table 2A calculation of $\ln [\sinh (\alpha \sigma)]$, $\alpha = 0.058$ for the 7075Al alloy

<table>
<thead>
<tr>
<th>strain rate(s$^{-1}$)</th>
<th>250°C</th>
<th>300°C</th>
<th>350°C</th>
<th>400°C</th>
<th>450°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8</td>
<td>6.41</td>
<td>5.57</td>
<td>4.64</td>
<td>3.25</td>
</tr>
<tr>
<td>0.1</td>
<td>7.43</td>
<td>5.63</td>
<td>4.24</td>
<td>3.31</td>
<td>1.97</td>
</tr>
<tr>
<td>0.01</td>
<td>5.68</td>
<td>4.24</td>
<td>2.78</td>
<td>2.14</td>
<td>1.01</td>
</tr>
<tr>
<td>0.001</td>
<td>4.53</td>
<td>3.31</td>
<td>2.2</td>
<td>1.38</td>
<td>-0.02</td>
</tr>
<tr>
<td>n</td>
<td>1.83</td>
<td>2.13</td>
<td>2.26</td>
<td>2.07</td>
<td>2.4</td>
</tr>
</tbody>
</table>

$$S_d = \sqrt{\frac{1}{N} \sum_{i=1}^{N} (n_i - \bar{n})^2}, \ N = 5 \ i = 1 - 5, \bar{n} = 2.02 \ S_d = 0.117$$
2. Calculation of strain hardening rate

Strain hardening rate can be expressed as $$\frac{\delta \sigma}{\delta \varepsilon}|_{\dot{\varepsilon}T}$$, while the relationship between stress and strain is defined as $$\sigma = f(\varepsilon)$$, where $$\sigma$$ is true stress and $$\varepsilon$$ is true strain. It can be seen from the flow curve that once the yield stress is reached, the material exhibits rapid strain hardening which decreases as deformation progresses. Strain hardening rate in a rapid hardening region at low strain is considered as initial strain hardening rate. It was evaluated by $$\frac{\Delta \sigma}{\Delta \varepsilon}$$ at low strain (0.01-0.03) in a small increment of $$\Delta\varepsilon$$. $$\Delta\varepsilon$$ was chosen to be 0.005 in the present work. The schematic diagram for the determination of strain hardening rate value is shown in the following Figure:

![Figure 1A schematic diagram for the calculation of strain hardening rate](image-url)
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Publication