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Deformation behaviour and microstructural development of ceramic particles reinforced 6061 aluminium alloys [microscript]

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DEFORMATION BEHAVIOUR AND MICROSTRUCTURAL DEVELOPMENT OF CERAMIC PARTICLES REINFORCED 6061 ALUMINIUM ALLOYS

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

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The University of Wollongong

By
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Department of Materials Engineering

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ABSTRACT

The deformation behaviour of 6061 aluminium reinforced with particulate Al₂O₃ was studied by cold working as well as by high temperature compression. In addition to deformation studies, the ageing behaviour of the composite was also examined by ageing the material at 125 and 170°C isothermally. It was found that peak hardness appeared much earlier in reinforced alloy than in monolithic alloy when the materials were aged at 170°C suggesting that the existence of reinforcement could accelerate ageing. Studies showed that β" phase (or called GP-II zone) was the only hardening agent in 6061Al alloys, and changes in morphologies of β" phase would result in variation in hardness. During ageing, β" phase started appearing in a short needle-shape, then fine needle shape which gave the samples' highest hardness, and as ageing continued, the β" phase became coarse and the hardness of the alloy decreased. Observations using TEM and differential scanning calorimeter (DSC) indicated that formation and coarsening of the β" phase (or called GP-II zone) were accelerated in the reinforced alloys. This acceleration is probably due to high diffusivity resulted from high density of dislocation and elastic strain field generated by thermal mismatch between the reinforcement and the matrix. However, there was little difference in ageing response for reinforced and unreinforced alloys when they were aged at 125°C. This is because at low temperatures, formation and coarsening of the β" phase are very slow, and the effect of the reinforcement is not obvious. The composite and the matrix alloy were also aged in a temperature range of 125 to 350°C for one hour and it was found that accelerated ageing occurred in the composite during isochronal ageing.

To investigate the effect of straining on ageing behaviour of the composite at low temperature, the material was given 55% reduction prior to ageing, and then was aged at
125°C for up to 780 hours. It was found that deformation prior to ageing could diminish formation of \( \beta'' \) zone and degrade ageing ability of the material. For comparison, SiC particle reinforced 6061Al alloy was also aged under identical conditions and it was found that SiC reinforced material exhibited slightly faster ageing response compared to \( \text{Al}_2\text{O}_3 \) composite. This difference in the two composites seems to be due to higher dislocation density and/or internal strain caused by considerable difference in coefficients of thermal expansion between SiC and the matrix compared to \( \text{Al}_2\text{O}_3 \) composite.

In order to examine the deformation characteristics of these materials after cold working and on subsequent heat treatments, the \( \text{Al}_2\text{O}_3 \) reinforced 6061Al alloy and the matrix alloy were given reductions of 35, 55, and 70% at room temperature and then annealed in a temperature range of 200 to 500 °C for one hour. The results showed that, in the cold-worked state, the presence of large particles reduced the microband width and increased the dislocation density and hardness. During annealing, the large particles acted as nucleation sites for recrystallization, especially closely located particle groups which have a strong capability to stimulate recrystallization. Nucleation of recrystallization in the composite was dominated by large alumina particles resulting in a lower recrystallization temperature in comparison to the matrix alloy. Isochronal annealed samples (at 300°C after given 55% reduction in both the composite and the matrix alloy) showed that the recrystallization kinetics in reinforced material was accelerated due to the presence of large particles.

To investigate the effect of solute and precipitation on recrystallization behaviour, both reinforced and unreinforced alloys were heat treated in T4 condition, and then were given 55% cold reduction followed by isochronal annealing. It was found that the materials showed much higher dislocation density than O tempered sample (OT) due to solute drag during cold deformation. On subsequent annealing, recrystallization occurred
at a higher nucleation rate leading to a finer grain structure in the materials in T4 condition.

Although the degree of cold deformation and annealing temperature did have some influence on recrystallized grain size, the interparticle spacing of Al₂O₃ was found to be the main controlling factor affecting grain size in the composite. Results indicated that reinforced material did not show significant grain growth even after annealing at 540°C for 40 hours in a sample given 55% cold reduction compared to the monolithic alloy.

The hot deformation behaviour of Al₂O₃ composite was investigated using uniaxial compression. Both reinforced and unreinforced 6061 aluminium alloys were deformed in a temperature range from 200 to 500 °C and at constant strain rates of 0.001 to 1 s⁻¹. The samples were water quenched following a true strain of 0.9 at any particular testing temperature. The flow stress at 200°C was the only one which exhibited strain hardening during testing but the true stress-true strain curves of the materials deformed in a temperature range from 250 to 500°C were of the type which showed that dynamic recovery was occurring during deformation. The results showed that, at low temperature, the composite displayed much higher proof stress and strain hardening rate than that in the monolithic alloy. As the temperature increased, the strength of the composite decreased at a higher rate compared to the matrix alloy.

Deformation behaviour of the materials at elevated temperatures was examined by evaluating the stress exponent and activation energy for deformation. It was found that the reinforced alloy exhibited higher values of stress exponent (n') and activation energy for deformation (Q) compared to the monolithic alloy indicating that the composite behaved similarly to that shown by oxide dispersion hardening alloy at high temperatures. However, the values of n' and Q for the present composite are not
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extremely high compared to other studies reported.

The microstructures of the alloys deformed at temperatures below 300°C consisted of fine substructures with a high density of dislocations and relatively small subgrain size. However, above 350°C the subgrain boundaries became sharper and the density of dislocations decreased considerably. When deformed at 500°C, both reinforced and unreinforced alloys displayed large well developed subgrains of a size up to 7 μm for the composite and 10 μm for the monolithic alloy. Substructure size was found to be inversely related to both lnZ (Z is temperature compensated strain rate) and the flow stress. A much finer substructure was present in the reinforced material compared to the matrix alloy under same deformation conditions suggesting that the presence of the large particles in the reinforced alloy could be responsible for refining the microstructure and thus leading to higher strength in the composite.

The effect of pre-treatment on deformation behaviour was studied by comparison of flow stresses and structural development in the composite with OT and T4 conditions. The material with T4 condition displayed inconsistent flow stress during straining, that is, the stress reached to peak value in the beginning of plastic straining then decreased as straining continuing. This phenomenon was obvious when the composite was deformed at temperatures below 350°C. Above 400°C, the flow curves were flat. The microstructure study showed that deformation was also dominated by dynamic recovery in the material with T4 condition and decrease of stress with straining was found to be due to occurrence of dynamic precipitation during deformation.

The effect of high temperature annealing on the hot worked structure was studied. Hot worked samples were annealed at 500°C for one hour, and the recrystallized structure was examined. The results showed that the samples deformed at low temperatures and
high strain rates exhibited fully recrystallized structure after annealing, but as the deformation temperature increased to a critical level, no recrystallization occurred during annealing in the hot deformed samples. The critical temperature was dependent on strain rate. As strain rate increased, critical temperature decreased. Both reinforced and unreinforced materials showed similar behaviour. Three dimension diagram (between $\dot{\varepsilon}$, $T$, and $d$) showed that there were two regions in the recrystallized grain size distribution depending on the deformation temperature and strain rate in reinforced and unreinforced materials. Fine grain size was obtained in low temperature and high strain rate region, whilst coarser grain structure existed in high temperature and low strain rate region. Low nucleation rate was considered to be the reason for the formation of large grains during annealing in these hot worked samples.
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Table 5.3 Recrystallized grain sizes ($\mu$m) in $\text{Al}_2\text{O}_3/6061\text{Al}$ composite (and 6061Al alloy) deformed at different temperatures and strain rates followed by annealing at 500°C for an hour.

Table A-1 Calculation of $\ln \left[ \sinh(\alpha \sigma) \right]$, $\alpha=0.03$ for $\text{Al}_2\text{O}_3/6061\text{Al}$ composite.

Table A-2 Calculation of $\ln \left[ \sinh(\alpha \sigma) \right]$, $\alpha=0.035$ for 6061Al monolithic alloy.
LIST OF SYMBOLS

\( \dot{\varepsilon} \) : Constant strain rate \( (s^{-1}). \)

\( \alpha \) : Constant.

\( A_1, A_2, A_3 \) : Constants.

\( \beta'' \) : One stage of the GP zones in 6061 aluminium alloy.

\( \beta' \) : One form of the precipitates prior to \( \text{Mg}_2\text{Si} \).

\( d \) : Subgrain size \( (\mu m) \), \( (d_c \text{ for the composite, and } d_m \text{ for the monolithic alloy}) \).

\( \Delta \varepsilon \) : Increment of true strain.

\( \Delta \sigma \) : Increment of true stress.

\( \varepsilon \) : True strain.

\( H_0 \) : Hardness of as deformed material.

\( H_i \) : Instantaneous hardness.

\( H_r \) : Hardness of recrystallized material.

\( I_r \) : Restoration index, \( I_r = \frac{H_0 - H_i}{H_0 - H_r} \).

\( K \) : Constant.

\( m \) : Strain rate sensitivity.

\( n' \) : Stress exponent.

\( n_1 \) : Temperature independent constant.

\( Q \) : Activation energy for deformation \( (kJ/mole) \).

\( R \) : Gas constant.

\( \sigma \) : True stress \( (MPa) \) (flow stress, \( \sigma_c \text{ for the composite and } \sigma_m \text{ for the monolithic alloy}) \).

\( \sigma_{0.2} \) : 0.2% proof stress.

\( T \) : Deformation temperature \( (K) \).

\( Z \) : Temperature compensated strain rate \( (\text{Zener-Hollomon parameter}) \).
CHAPTER 1

INTRODUCTION

1-1 Evaluation of Discontinuous Fiber and Particle Reinforced Metal Matrix Composites

1-1-1 Discontinuous Metal Matrix Composites (DMMCs)

The metal matrix composites (MMCs) can be categorized in three groups which are: 1. dispersion-strengthened MMC; this composite is characterized by a matrix alloy containing uniformly dispersed particles. The particle size ranges from about 0.01 to 0.1 \( \mu \text{m} \) in diameter, and the volume fraction of particles ranges from 1 to 15%; 2. particle reinforced MMC; this composite is identified by dispersed particles of greater than 1.0 \( \mu \text{m} \) in diameter with a volume fraction of 5 to 40%; 3. fiber or whisker-reinforced MMC; whiskers range from 0.1 to 250 \( \mu \text{m} \) in length, and long fiber may be produced as long as continuous fiber.

However, MMCs can also be classified in to two other categories, these are (i) continuous fiber reinforced composite, and (ii) discontinuous fiber (whisker) or particle reinforced composite (2). These two types of composites are quite different not only in the way they are manufactured and thermomechanically processed, but they are also quite different in their strengthening mechanism and their mechanical properties (2-8).

Metal matrix composite materials have been developed over a period of about 30 years. However, the initial emphasis was on continuous filament MMCs (1,2,7). The
cost of producing and manufacturing these materials is high. Even though new low-cost fibers have been developed, the materials are still quite expensive to produce and difficult to manufacture compared to discontinuous fiber or particle reinforced composites (1-9).

The application of continuous fiber reinforced metal matrix composites is limited by their high cost and poor workability. Therefore, in recent years, DMMCs have been developed and investigated in detail, especially aluminium matrix composites. The prime advantage of discontinuous aluminium matrix composites is that they can be formed by conventional metal working-processes such as rolling, forging, extrusion, machining, and swaging (1,2). Obviously, it is also cheaper to make ceramic whisker, particle, or platelet than produce continuous fiber. These advantages give discontinuous MMCs the potential to be advanced commercial engineering materials.

The most prominent discontinuous reinforcements used are SiC, Al₂O₃, Si₃N₄, and B₄C in both whisker and particulate form (1-6,8-16). These ceramic fibers or particles possess high strength, modulus, and hardness (15) and can be combined with the matrix alloy by casting, powder processing, and deposition processing in volume fraction ranged normally from 5 to 30% (1-6). Aluminium based alloys are considered to be the most common matrix alloys used for discontinuous MMCs (1,2,5,6,8-16). Studies show that the strength and modulus of aluminium alloys could be increased significantly by adding discontinuous fibers and particles (16-19). The following section will review the strengthening mechanism in the discontinuous MMCs, especially particulate MMCs.
1-1-2 Strengthening Mechanism of DMMCs

The strengthening mechanism of discontinuous MMCs is different from that of continuous MMCs. In continuous MMCs, the prime role of the fibers is to carry the load, while the metal matrix serves to transfer and distribute the load to the fibers. If assuming high interface efficiency, the mechanical properties of the composite depend more on the properties of the fiber than the properties of the matrix (1). The strength of continuous fiber reinforced composites could be described simply by a law of mixture (1,7,20). The stiffness of the composites is expressed as

\[ E_c = V_f E_f + V_m E_m \]  

and flow stress can be written as

\[ \sigma_c = V_f \sigma_f + V_m \sigma_m \]

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

However, short fiber or particle can not fully carry the load because they are discontinuous. Therefore, law of mixtures tends to give poor estimates for the stiffness and tensile strength of short fiber and particle reinforced MMCs. Alternatively, several other models were developed to predict mechanical properties of discontinuous MMCs, such as the shear lag model, Eshelby's model and their modified models which are based on a continuum approach (15, 21-23). The strengthening mechanism based on dislocations was suggested by Arsenault and other
researchers (8, 10, 24-30). It was considered that the composite properties could be quite well modelled using dislocation theories. Humphreys and Miller described the strengthening mechanism of the discontinuous MMCs as similar to that of conventional two-phase alloy (31-34). The later two mechanisms are based on microstructural change in the matrix caused by introduction of the reinforcement. These strengthening mechanisms will now be discussed in the following section.

1-1-2-1 Strengthening Mechanisms of DMMCs Based on a Continuum Approach

Typical models based on a continuum approach are the shear lag model and Eshelby's model. The shear lag model was originally developed by Cox (21). It was suggested that the discontinuous fibers were uniaxially aligned in the matrix, and the composite was load in a direction parallel to the fibers. The load in the fiber would be

\[ P = E_f A e \left( 1 - \frac{\cosh \beta (L/2-x)}{\cosh (\beta L/2)} \right) \]  

where \( E \) is the modulus of the fiber, \( A \) is the area of section of the fiber, \( e \) is a strain in the composite, \( L \) is length of the fiber, and \( \beta = (H/E_f A)^{1/2} \), \( H \) is a constant which depends on the geometrical arrangement of the fibers. Kelly summarized and derived tensile stress in the fiber according to Cox's model (15), \( \sigma = (P/A) \)

\[ \sigma_f = E_f e \left( 1 - \frac{\cosh \beta (L/2-x)}{\cosh (\beta L/2)} \right) \]  

Equations 1-3 and 1-4 were derived in the end condition of \( P = 0 \), that is tensile stress can not be transferred at fiber ends (15, 21). This is a conventional shear lag model. Nardone and Prewo modified this model by considering the tensile stress transfer at fiber ends, and gave a satisfied prediction of strengthening effect of both SiC whisker
and platelet materials (35). When applying boundary conditions, $\sigma_f = \text{constant} \ (\sigma_0)$ at $x=L/2$ and $d\sigma/dx=0$ at $x=0$, the stress field in the fiber can be given by (1)

$$\sigma_f = E_f \varepsilon \left\{ 1 + \left[ \frac{((\sigma_0/E_f)-1) \cosh \beta x}{[\cosh(\beta L/2)]} \right] \right\} \quad 1-5$$

This model is best suited for an aligned short fiber composite where short fibers of uniform length and diameter are all aligned in the loading direction and distributed uniformly throughout the material. This is because the model is based on one dimensional elastic field (15,35). However, in the most whisker or particulate composites, short fibers or particles are aligned optionally in three dimensions, and the aspect ratio of the reinforcement becomes small. Subsequently, shear lag models tend to give poor estimates for the mechanical properties of a composite.

To solve the three dimensional problem in the composite, Eshelby's model was introduced and modified to predict strengthening of the composite (1, 22, 23). It is considered that Eshelby's model is more suited for prediction of short fiber and particle reinforced composites (36). The model proposed a method to solve three-dimensional elasticity problems where inclusions or inhomogeneities of ellipsoidal shape are embedded in an infinite elastic matrix (1, 22, 23). The basic idea of the model is to calculate stress field both within and outside the inclusion or inhomogeneity by calculating the elastic tensors and the tensors that are related to the geometry of the inclusion and the matrix Poisson’s ratio. The stress field inside inhomogeneity is given by

$$\sigma_{ij} = C_{ijkl}(e_{kl}^* - e_{kl}) \quad 1-6$$
where \( C_{ijkl} \) is elastic modulus tensor of the matrix, \( e_{kl} \) is the total strain and related to displacements and eigenstrain \( e_{mn}^* \) by

\[
e_{kl} = S_{klmn} e_{mn}^*
\]

where \( S_{klmn} \) is 'Eshelby's tensor 'which is a function of the geometry of the inclusion and the Poisson's ratio when the matrix is isotopic. By comparison, Eshelby's model is more complex than the shear lag model but it gives better approximation in predicting the strength of the composite. Other models based on variational principles have been reviewed and summarized by Mura (23), Taya and Arsenault (1, 36) such as the self-consistent models. Since those models contain some unknowns and complex tensors for evaluation of the stress field, great numerical calculations are required via the use of computer.

Those models mentioned above are described as the models of composite strengthening or continuum models (1). They are mostly based on conventional elastic stress-strain analysis in which the reinforcement and the matrix 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.

It was generally found that the agreement between theory and experiment was quite satisfactory for the case of well-aligned long fibers (1,2,9,15, 20-23). However, several studies have shown that those models based on a continuum approach failed to predict strengthening of the materials containing short fibers or particulates under certain conditions (8,10,37). It was found that the observed strengthening due to the reinforcement was greater than that predicted by the continuum models. Arsenault and Fisher considered that the likely cause of most of the discrepancies is
that the matrix material itself is strengthened by the presence of the SiC fibers or particulates in the composite. Such strengthening of the metal matrix could result from a higher dislocation density and finer substructure than in monolithic alloy (10). Yang and Pikard et.al. also found that the discrepancies between calculation using continuum approach and experiment results for the as-cast particle reinforced composites appear to be related to a higher matrix strengthening caused by precipitation (35). Therefore, it is considered that strengthening of the composite is not only dependent on composite strengthening which is described by continuum approach but also relays on strengthening of the matrix caused by the reinforcement, especially for the short fiber or particulate composites (8,10,24,25). Strengthening mechanisms based on strengthening of the matrix or a micromechanism approach will be reviewed in next section.

1-1-2-2 Strengthening Mechanism Based on Strengthening of the Matrix

Typical strengthening mechanisms are based on the dislocation strengthening mechanism suggested by Arsenault and other researchers (8,10,25,27-28), and the micromechanical mechanism described by Humphreys (31-33).

Dislocation strengthening mechanism was introduced to property analysis of MMCs by many researchers (1,8,10,24,25), and, according to this mechanism, the discontinuous composites are strengthened by:

- High dislocation densities due to dislocation generation as a result of differences in coefficients of thermal expansion between the reinforcement and the matrix
- Small subgrain size as a result of the generation of a high dislocation density
- Residual elastic stresses
• Differences in texture
• Classical composite strengthening (load transfer)
• Dispersion strengthening

High dislocation density and small subgrain size are considered to have more effect on the strength of the composite. A model based on dislocations generated by mismatch of the thermal expansion coefficients between the reinforcement and the matrix was derived by Arsenault and Shi (1, 8, 28) as following:

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

where \( \Delta \sigma \) is the increase of yield strength, \( \alpha \) is a geometric constant, \( \mu \) is shear modulus of the matrix, \( V_r \) is the volume fraction of the reinforcement, \( b \) is the Burgers vector, \( B \) is a constant ranged from 4 to 12 according to the aspect of the ratio \( l/d \) of the reinforcement, and \( \varepsilon \) is misfit strain and \( t \) represents the smallest dimension. This model was derived from a schematic diagram of the particle and punched dislocations (28). If the morphology of the reinforcement is considered, the equation 1-8 will be rewritten as

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

where \( R \) is the aspect ratio of the reinforcement, when \( R<1 \), it represents whisker morphology, when \( R>1 \), it is a platelet morphology, and when \( R=1 \), it is a representation of equiaxed particle. \( V \) is volume of single reinforcement (1). It was found that the experimental data agreed with theoretical prediction when the particle size was small, but the discrepancy was large when the particle size was large (1, 8, 28). This was considered due to shorter edge length over surface area ratio of the large
particle compared to small particle. The prediction of dislocation density based on prismatic punching which generates dislocation by the edge of the particle is lower than the dislocation generated by particle surface, so called secondary dislocation generation (1).

Strengthening mechanisms based on the micromechanism approach discussed and summarized by Humphreys are as following (31, 32):

- Quench strengthening
- Orowan strengthening
- Grain strengthening
- Sub-grain strengthening
- Work hardening

Quench strengthening was considered to mainly resulted from dislocations generated by mismatch of the thermal expansion coefficients between the reinforcement and the matrix. It depends on reinforcement size (d), volume fraction (Fv), mismatch of thermal expansion (ΔC), and temperature change (ΔT). The strength (σq) can be expressed as

$$\sigma_q = \alpha G b (12 \Delta T \Delta C F_v / bd)^{1/2}$$

where G is the matrix's shear modulus, b is the Burger's vector and \( \alpha \) is a constant between 0.5 and 1.
Orowan strengthening was only considered when the reinforcement is smaller than 1µm. Therefore, it is not applicable to the most of the composites that contain particles much larger than 1µm in size.

Grain strengthening was caused by fine grain size in the composite since the grain could be refined during thermomechanical processing through high recrystallization nucleation rate stimulated by reinforcement. If it is assumed that each particle nucleates one grain, the grain size after recrystallization will be given by:

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

1-11

D is normally smaller than the grain size of unreinforced alloy, and it may contribute to the yield strength of the composite.

Work hardening of the MMCs was considered not only to be caused by quenched dislocation structure, but also results from load transfer by means of Orowan loops, and geometrically necessary dislocations created by the reaction between the reinforcement and the matrix during high strain deformation (32, 37, 38).

Comparison was made between calculation and experimental data using Humphreys' approach (32). It was found that prediction of the strength depended on particle size. For large particle composite, the strength was underestimated by the theory but for small particle reinforced composite, it was overestimated (32).

It can be noticed by comparing the statement of Humphreys with that of Arsenault that they both consider that the most significant contribution to the strength of simple particulate MMCs are the strengthening of the matrix, even though they put emphasis
on different aspects, and approached strengthening mechanisms in different ways (8, 10, 25, 26, 31-34). Arsenault suggested that strengthening of the composites is due to a nonconventional phenomenon, that is, the increase in dislocation density and decrease in the subgrain size caused by the mismatch of the thermal expansion coefficients between the reinforcement and the matrix (8,10, 26). Humphreys described the mechanical behaviour of the composite using a micromechanical approach based on dispersoid strengthening, and suggested that high density of dislocation in the composite is caused by quench or deformation, and work hardening was considered as important as dislocation mechanism on strengthening of the composites (31, 32,34).

By summary, these strengthening mechanisms described by Arsenault and Humphreys et. al. consider that the matrix microstructure dominates the mechanical properties of the composites (8, 10, 24-26, 31-34). Therefore, it is necessary to study the effect of the reinforcement on microstructure changes, and the relationship between mechanical behaviour and structural development. Among these structural phenomena of the matrix, high density of dislocations is a most important factor for the strengthening. The interaction between the reinforcement and the matrix, and generation of the dislocations will be discussed in the following section.

1-1-3 Generation of the Dislocation by the Particle

The early study of the dislocation generated by a misfitting particle and a ductile matrix was carried out on observation of reaction between precipitate or inclusion and dislocations during the 1950's and '60's. One of the examples was the study conducted by Eikum and Thomas, in which they found that the nucleation of dislocations from precipitates can be expected to occur in view of large stresses set up
due to the volume changes accompanying precipitation since the stresses associated with the formation of precipitates can be relaxed by prismatic punching of dislocation loops (39). Weatherly employed prismatic punching mechanism and Eshelby's theory (23) to explain the transformation of full coherency to partial coherency of precipitates (40). The misfitting strain, so called constrained strain, and shear stresses generated by the misfitting particles with different morphologies was investigated. It was suggested that nucleation of the dislocation depended on the magnitude of the strain and shear stress in the matrix generated by misfitting particle.

An intensive study on the generation of dislocation at misfitting particles in a ductile matrix was carried out by Ashby and Johnson (41). It was considered that an energy barrier $\Delta E^*$ must be overcome in order to nucleate a dislocation. $\Delta E^*$ is related to constrain strain caused by misfit, and it decreases as the misfit at the particle increases. With a larger misfit, the energy of the system is decreased considerably by dislocation generation. In another study, Ashby, Gelles, and Tanner discussed the stress at which dislocations are generated at a particle-matrix interface in two copper alloys containing $\text{SiO}_2$ and $\text{Al}_2\text{O}_3$ particles (42). It was found that when a hydrostatic pressure is applied to a metal containing particles or inclusions of different elastic constant, the local shear stresses can be large enough to cause the interface to emit dislocations. The critical pressure at which dislocation is generated depends on the particle size. As particle size increases, the critical pressure or shear stress for dislocation generation decreases. This means that strength of the material decreases as particle size increases, but it is easier to generate dislocation at larger particles. These studies carried out by Ashby et al have attributed to the basic principle of generation of dislocation by misfit of the particle in plastic matrix.
In composites, most reinforcements are much larger than those precipitates or inclusions described by Weatherly and Ashby. The misfit between the reinforcement and the matrix could be quite large. The misfit can be introduced by a mismatch of the thermal expansion between the reinforcement and the matrix or by plastic deformation of the composite. This misfit is large enough to generate dislocations according to Weatherly and Ashby's theories (40-42).

In the most of the composites, reinforcements have different physical properties from the matrix. For instance, most reinforcements have a much smaller thermal expansion coefficient than the matrix (43). When the reinforcement is strongly bonded within the matrix, the difference in shrinkage of the two materials during cooling from high temperature will generate misfit strain, and a stress field will be built up at the interface. The stresses may only be relaxed by diffusion or by dislocation nucleation followed by dislocation glide and climb (30,44). Diffusion normally occurs at high temperature, and then dislocation nucleation can be expected.

One systematic study of dislocation distribution in the composite was carried out by Chawla and Metzger using etch-pit technique (45). It was found that the dislocation density rises to high values toward the fiber-matrix interface. The study showed that this increase of dislocation density was caused by mismatch of thermal expansion between tungsten fiber and copper matrix. The misfit strains in three directions were calculated according to the thermal expansion coefficients of W fiber and copper matrix. Then the principal stresses in the matrix were calculated. It was found that only a 1°C temperature change would have been able to induce shear stresses up to 100 gm/mm², more than sufficient to produce plastic flow of the matrix the micro-yield stress of which was 50 gm/mm² at room temperature.
In the study of Ni-W eutectic composite, Williams and Garmong found that the dislocations in the matrix are largely concentrated around the W fibers, and appear to have been emitted from the region near the fiber/matrix interface (46). It was considered that these dislocations were generated by thermal stresses during quenching.

A direct observation of dislocation generation at Al/SiC interfaces was made by Vogelsang, Arsenault, and Fisher in an in-situ HVEM study (47). They observed that, at high temperature, the matrix was free of the dislocation near the SiC fiber. As the temperature decreased, dislocations first appeared around the fibers, and dislocation density increased as the temperature continuously decreased. To predict dislocation density generated by thermal mismatch in whisker and particle reinforced composites, Arsenault and Shi developed a simple model based on prismatic punching (28). The model is as following:

\[
\rho = \frac{B V f \epsilon}{b(1-V_f)} t^4
\]

where \( B, V_f, b, t, \) and \( \epsilon \) have been defined in equation 1-8. It can be seen that the intensity of dislocation generated at the Al-SiC interface is related to volume fraction, size, and shape of the SiC particles. It was considered that with constant volume fraction, the average dislocation density decreased as the particle size increased. Also the intensity of dislocations generated is much greater at the corners of the particle. Cottu and Couderc also found that the dislocation density was larger in the vicinity of fiber end than the middle of the fiber length (48). It is considered that the strain generated by misfit is greatest at the corner of the particle or the end of the fiber (47).
Besides the dislocations generated by thermal stress, deformation also can introduce additional dislocations in the composite. These dislocations are called "geometrically necessary" dislocations by Ashby according to the plastic bending model described by Cottrell (49, 50). The dislocations occur in order to allow compatible deformation of a system with geometrical constraints such as hard particles that do not deform as the surrounding ductile matrix. The density of geometrical necessary dislocations, \( \rho^G \), is given by:

\[
\rho^G = \frac{4\gamma}{b l}
\]

where \( l \) is the interparticle spacing, and \( \gamma \) is the shear strain. It can be seen that the dislocation density is directly related to the amount of shear strain and interparticle spacing, that is, as the deformation increases or interparticle spacing decreases, the density of the geometrical necessary dislocations increases. For a 1% shear strain and \( l=4 \mu m \) in a Al matrix, \( \rho^G \) equals approximately \( 3 \times 10^{13} \) m\(^{-2} \) (47). This additional dislocation density will significantly affect both the strength and the microstructural development of the composite. In the next sections, microstructural development in alloys containing particulate or fibre reinforcements during thermomechanical processes will be discussed.
1-2 The Effect of Reinforcement on Age Hardening Behaviour of Aluminium Composites

1-2-1 Acceleration of Age Hardening due to Presence of the Reinforcement in Composites

As reviewed above, the presence of the non-deformable short fibre or particle could increase density of dislocation and elastic strain in the matrix of the composite. These changes in the structure of the matrix may not only affect properties of the composites but also affect thermomechanical processes subsequently. One of these processes is ageing. Early studies carried out by Nieh and Karlak in B₄C reinforced 6061Al alloy (51), and Rack in SiC whisker reinforced 6061Al composite (52) showed that the addition of the reinforcements to 6061Al alloy could accelerate its ageing response. However, the most extensive evidence for the effect of a fine dispersed second phase on age hardening is the work of Ceresara and Fiorini on sintered aluminium powder (SAP) alloys (53,54). From the hardness and the resistivity measurements on (Al-Mg-Si)-Al₂O₃ and (Al-Cu)-Al₂O₃ alloys, Ceresara and Fiorini observed that presence of 3% of Al₂O₃ particles could inhibit G P zone formation and slow down natural ageing. During artificial ageing these SAP alloys could be prematurely over-aged resulting in reduced age hardening compared to the conventional powder materials. These studies indicate that the heat-treatment response of an alloy containing fine ceramic particles or whiskers could be significantly different from that of the unreinforced alloy.

Alternative of ageing behaviour in the reinforced materials was considered to be caused by several factors. Firstly, the quenched-in vacancies following solution treatment in the reinforced materials reduced (55-57). This lack of quenched-in vacancies was
considered due to the availability of large numbers of vacancy sinks at the reinforcement-matrix interfaces (55,57), or the vacancy content was reduced by high density of dislocations that may provide more annihilation sites for retained vacancies (56). It was considered that the lack of quenched-in vacancies in the reinforced alloy could inhibit its natural ageing due to the suppression of GP zone formation, and could also decrease its hardening level during artificial ageing (55,57). Secondly, high density of dislocations generated from the thermal mismatch between the reinforcement and the matrix during quenching, and the presence of a high diffusive interface in the reinforced materials could accelerate their ageing response (51, 56, 58-70). Moreover, it was also reported that residual stresses or elastic strain generated by the reinforcements may accelerate ageing in the composites (59,62,71). These two factors could affect ageing behaviour simultaneously resulting in complicated ageing response in the reinforced materials. The work conducted by Friend et al. showed that GP zone formation was suppressed due to the lack of the quenched-in vacancies in δ-alumina fiber reinforced 6061Al alloy, and the composite loosed its age hardening level (55,57). Recently, Dutta et al. also suggested that the addition of alumina reinforcements has a more profound effect on the ageing behaviour of age hardenable aluminium matrix composites than just the acceleration of precipitation kinetics because reinforcement addition decreases the vacancy concentration in the matrix (62). However, several researches recently have shown that presence of the reinforcement in the alloy does not decrease its age hardening level, but accelerate ageing process (51,52,55, 58-75).

Acceleration of ageing was considered mainly resulted from high density of dislocations and/or internal stresses generated by the reinforcement (51,55,58-75). It is well known that dislocations act as the sites for heterogeneous nucleation during precipitation (76,77). It was found that precipitate nucleation in the composite matrices
occurs on two kinds of sites: dislocations generated as a result of the thermal mismatch between the reinforcement and the matrix and quenched-in vacancy loops. The nucleation on the first type of sites was found to be accelerated (62). The increased dislocation density enhances the nucleation of strengthening precipitates whereby the incubation time for precipitate nucleation and the ageing time to achieve the peak hardness in the matrix are significantly reduced for the composites compared to monolithic alloys (70). The dislocations could also act as paths for increased atomic transport during precipitates growth, called pipe diffusion (78, 79). Therefore, growth of the precipitates in the reinforced materials was enhanced (62).

1-2-2 Ageing Behaviour of 6061Al Alloy

6061Al alloy is an age hardenable alloy containing mainly elements Si and Mg (80). Even though the precipitation in this alloy or Al-Si-Mg alloys has been studied for many years, ageing sequence is still not properly defined (51, 81-88). It is necessary to review the ageing sequence in 6061Al alloy before effect of the reinforcement is discussed.

During 60's, Thomas (81) and Pasfley (82) found that the sequence of decomposition of the supersaturated solid solution in Al-Si-Mg alloy depended on ageing time and temperature and was continuous in the form:

\[
\text{Zones (needle-shaped)} \rightarrow \text{rods} \rightarrow \text{plates of the equilibrium Mg}_2\text{Si.}
\]

The orientation relationship between precipitates and the matrix was found to be \(\langle 100\rangle\text{needle zone} // \langle 100\rangle\text{matrix, and } \langle 110\rangle\text{Mg}_2\text{Si} // \langle 100\rangle\text{matrix} \) (81). Since the needles are aligned along the three cube axes of the aluminium matrix, they are
Fig. 1.1 Precipitates formed during ageing of an Al-Mg-Si alloy; a) GP-zones, b) $\beta'$-phase, and c) $\beta$-Mg$_2$Si (89).
coherent with the matrix, and give rise to faint streaks on the selected-area diffraction patterns (82). It was also found that the stage needle→rods→plates are continuous, so that the equilibrium Mg$_2$Si phase can be formed by only minor atom movements within the rods.

The structure of the precipitates in Al-Si-Mg alloy was studied by Jacobs in more detail (83). The study confirmed that ageing sequence is, needle-shaped zones→rods(β')→Mg$_2$Si(b). The rod-shaped precipitates have a hexagonal unit cell had an orientation relationship with the matrix, (001)$_{\text{p7}}$//(100)$_{\text{matrix}}$, and [100]$_{\text{p7}}$//[011]$_{\text{matrix}}$. Jacobs displayed morphologies of three stage precipitates in the first time shown as Fig.1.1(a-c).

The work carried out by Rack gave out more detail information about ageing sequence in 6061Al (Al-Si-Mg) alloy (84). The sequence appeared to involve the following: supersaturated solid solution→vacancy silicon clusters→GP zones→disordered needle-shaped phase→ ordered needle-shaped phase→ hexagonal rod-shaped phase (a=7.05Å, c=4.05Å)→hexagonal rod-shaped phase(a=7.05Å, c=4.05Å)→equilibrium β-Mg$_2$Si platelets. It could be summarized to:

Silicon cluster→GP zone→needle-shaped phase→rod-shaped phase→equilibrium Mg$_2$Si plates (β).

Rack 's work made a big progress in identification of the ageing sequence in 6061Al alloy because silicon cluster and non-needle-shaped GP zone were identified, and development of needle-shaped phase was described. Lorimer reviewed others previous work on different aluminium alloys including Al-Si-Mg alloy and summarized precipitation sequence as;
Fig. 1.2 Development of precipitates during ageing of a 6061Al alloy at 200°C; a) GP-I zones after 100 seconds, b) needle-shaped GP-II zones or β" after 2000 seconds, c) rod-shaped β' phase after 50,000 seconds (62).
Supersaturated $\alpha \rightarrow$ needle-shaped zones $\rightarrow$ rod-shaped $\beta' \rightarrow \beta$ ($\text{Mg}_2\text{Si}$) (85).

These studies in precipitation and structural development in the alloy mentioned above were conducted using X-ray techniques and electron microscopy. When Differential Scanning Calorimetry (DSC) technique was introduced to precipitation analysis, it gave better understanding in kinetics and microstructural development of the precipitates (89-92). Studies of precipitation by DSC were carried out earlier in 7xxx and 2xxx alloys (89-92). Only recently, detailed calorimetric study of precipitation in 6061Al alloy was carried out by several researchers (56,62,88). In their studies, DSC thermograms were analysed corresponding to observation of TEM micrographs. Ageing sequence was identified and renamed as following:

Silicon cluster $\rightarrow$ GP-I zone $\rightarrow$ GP-II zone ($\beta''$) $\rightarrow$ rod-shaped $\beta' \rightarrow$ plate-shaped $\beta$-$\text{Mg}_2\text{Si}$

GP-I zone displays a dot shape, GP-II zone has a needle-shaped morphology, and $\beta'$ phase is rod-shaped as shown in Fig.1.2(a-c). It can be seen that GP-II or $\beta''$ phase has a similar morphology as needle-shaped phase in Fig.1-1a which was defined as zones (83,85). Two steps formation of the zones was firstly detected by Lutts using x-ray technique in 1961 (86). It described that first step of the zones possesses a high vacancy concentration, and the zones following show a periodicity along the segregate axis which lies parallel to one of the matrix cube directions. This was confirmed by later studies (62,84,88). Ageing sequence achieved by Dutta is quite similar to that explained by Rack, and it is a fairly detail description of the ageing sequence up to date in 6061Al or Al-Si-Mg alloy.
Hardening agent in 6061Al alloy has been reported to be needle-shaped zones (80, 86). From micrographs taken by Friend (55) and Dutta (62) in the peak-aged samples, it can be seen that needle-shaped zones (or called β" phase) are responsible for age hardening. However, some studies showed that the age hardening of 6061 is associated with the formation of the intermediate precipitate β'-Mg2Si instead of formation of the zones (57) or both (87). More work needs to be done in ageing sequence of the alloy by comparison of hardness measurement, TEM observation and DSC technique for determining hardening agent in this alloy.

1-2-3 Age Hardening Response in 6061Al Alloy Containing Particles

Early study of effect of reinforcement on ageing behaviour in Al-Si-Mg alloy was carried out by Ceresara and Fiorini in SAP-type (Al-Si-Mg)-Al2O3 composite (54). It was found that presence of oxide particles could reduce quenched-in vacancies in the matrix and subsequently reduce age hardening level. Fine grain and subgrain in SAP-type alloy caused by existence of Al2O3 could result in premature overageing in this alloy.

Nieh and Karlak studied ageing characteristics of B4C particle reinforced 6061Al composite in 1984 (51). The results showed that the addition of B4C particle to 6061Al alloy accelerates its ageing response. It was reported that this was due to the high dislocation density generated from the thermal mismatch between the reinforcement and the matrix, and the presence of a high diffusive interface in the composite. From then, many researches have been carried out to investigate ageing phenomenon in reinforced 6061Al or Al-Si-Mg alloys (52, 54-67).
These studies confirmed acceleration of the ageing response in reinforced materials, but several differences in the ageing characteristics were observed. First argument is that age hardening ability is reduced or increased. Some studies showed that age hardening ability was reduced, and overageing rate increased in the reinforced alloy because of lack of quenched-in vacancies and GP zones (54,55). Other researchers found that addition of the reinforcement increased the volume fraction of the GP zones, while suppressing the formation of the β' and β phases (62). Second argument is that the effect of reinforcement on the ageing kinetics of aluminium alloys depends upon ageing temperature (51-54,62). Rack found that the peak hardness in SiC<sub>w</sub>/6061Al occurs at earlier ageing times compared to monolithic 6061Al if ageing is carried out at 175°C, and at later times if aged at 125 °C (52). The work carried out by Bhagat et.al also indicates that acceleration of ageing in the 6061Al composite was more obvious at high ageing temperature than at low temperature (58). Esmaeili and Chawla also found this phenomenon in SiC reinforced 2014Al alloy (75). It was considered that, at low temperature, the nucleation of GP zones is mostly homogeneous and quenched-in vacancies play an important role in their formation. However, at high temperature above the zone solvus, no GP zones appear and the s' precipitates nucleate heterogeneously on dislocations directly in a Al-Cu-Mg alloy, and therefore, ageing in the composites is accelerated at high temperature (75). However, some studies show that acceleration of ageing in the composites is more favourable taken place at low temperature (51, 60). It was considered that, at low ageing temperatures, when both nucleation and growth is enhanced by presence of dislocations, dislocation density gradients raise the initial precipitation rate. However, at relatively high ageing temperatures, when lattice diffusion dominates and growth is not so dependent on dislocation distribution, accelerated ageing becomes less pronounced in composites (60). In the two statements above, both the studies showed that high density of dislocations is the main reason for acceleration of ageing in the
composites. The difference is that former considered that dislocation density has no effect on low temperature precipitation, and later considered it has because both nucleation and growth of early stage precipitation could be enhanced.

Another phenomenon shown in the aged composite is degradation of age hardening characteristics caused by chemical reaction between the reinforcement and the matrix. This was found by Friend et al. in $\delta$-Al$_2$O$_3$ whisker reinforced 6061Al composite (57). Munoz et al. also found degradation of ageing hardening in a $\delta$-Al$_2$O$_3$ fiber reinforced A356 alloy composite (61). It was found that decrease in ageing ability arises from chemical interactions between $\delta$-Al$_2$O$_3$ and the matrix which remove magnesium from the matrix and therefore suppress the formation of the age hardening intermediate precipitates.

1-2-4 Effect of Deformation on Ageing Characteristics of 6061Al Alloy and Its Composites

Various commercial practices utilize cold working in conjunction with aging, especially cold working prior to ageing: such as T3 temper (solution heat treating$\rightarrow$cold working$\rightarrow$natural ageing) and T8 temper (solution heat treating $\rightarrow$ cold working $\rightarrow$ artificial ageing) (93). It is well known that combination of deformation and precipitation could improve the properties of the aluminium alloys. This is not discussed in here, but the emphasis is put on effect of deformation on precipitation behaviour of 6061Al alloy.

An early study showed that deformation prior to ageing might be used to minimize the possible deleterious effects of precipitate free zones around grain boundaries because the dislocations generated by cold deformation may act as nucleation sites of
precipitation resulting in fairly uniform precipitation (94). This is considered as one of methods for improving properties of the alloy. Another effect of deformation on the ageing is that ageing sequence may be changed by deformation prior to ageing. This was studied in the work of Rack and Krenzer on 6061Al alloy (84). They have reported that prior strain could affect ageing sequence in a commercial 6061Al alloy. It was suggested that the principal effect of dislocation substructure generated by pre-strain lies in the control of the volume fraction, size and distribution of the needle-shaped phase. However, this effect is depended on the ageing temperature. It was found that ageing carried out at low temperature region where only cluster and GP zone formation occurred, the influence of the dislocation array on the precipitation reaction was little. If the ageing was conducted at high temperature region where the treatment involves the intermediate needle-shaped phase, the rate of precipitation appears to be enhanced (52,84,95). This could be explained as following; cold working increases density of dislocations, and these dislocations will act as nucleation sites of needle phase precipitation but not cluster or GP zone during following ageing since formation of the zones is determined by quenched-in vacancy which is normally distributed uniformly in the matrix. Therefore, the authors considered that cold working has very little effect on GP zone formation but will obviously accelerate heterogeneous precipitation of the needle-shaped intermediate phase (52, 84, 95).

Increase of the precipitation rate caused by pre-strain was also found in other studies (52,65,70,96). It is considered that cold work not only introduces dislocations and point defects into the alloys, but also increases the internal strain energy, which provides the driving force for microstructural changes occurred during aging. Christman and Suresh considered that increasing the dislocation density through cold working leads to the same accelerated ageing of the control alloy as is done by ceramic reinforcement (70).
A late study carried out by Rack in ageing of deformed and undeformed 6061Al alloy showed that maximum hardness in undeformed alloy was associated with formation of the rod-shaped β' phase. By contrary, in deformed alloy, maximum hardness associated with β' phase when ageing was conducted at low temperature, below the GP zone solvus. However, at high temperature, prior deformation tends to promote heterogeneous precipitation of the needle-shaped phase, with maximum hardness being associated with precipitation of the needle-shaped and β' phases (52).

Recently, precipitation behaviour of cold deformed 6061Al alloy and Al₂O₃ particle reinforced 6061Al composite was studied by Lee, Lu, and Chan using DSC technique (65). The study showed that the addition of Al₂O₃ particles in the alloy did not change its ageing sequence. Cold working, however, did change the sequence of the precipitation in both the monolithic 6061Al alloy and the composite.

The results obtained by Lee et.al are similar to that achieved by Rack in the way of that they both found out cold working could change ageing sequence in the materials (65). However, they have shown different ageing sequence in deformed alloys since Lee's results did not show any formation of needle-shaped β" phase in deformed alloys, but Rack did detect this needle-shaped phase, if it was a same phase as mentioned by Lee et al., which was considered as a result of heterogeneous precipitation on the dislocation generated by cold working (52, 65, 84, 95). More detail observation in microstructural development is needed for understanding effect of deformation on precipitation of the alloys.
1-3 Static Recrystallization Behaviour of Cold deformed Alloys Containing Particles

The effect of second phase particles on recrystallization of cold deformed metallic alloys have been studied for many years. Early work on the effect of particles was conducted in the dispersion-strengthened alloys. The particles mostly referred to dispersed or precipitated second phase instead of ceramic particles which are added to the materials. Only recent years, studies of effect of large ceramic particles on restoration behaviour of the composites were carried out. However, the theories for the microstructural development in the matrix of the composite were mostly based on studies of dispersion-strengthened alloys or precipitation hardened alloys. Therefore, to study restoration behaviour of the composites should first understand the behaviour of dispersions in the alloys.

1-3-1 Effect of Dispersed or Precipitated Particle on Static Restoration during Annealing

Many researches showed that presence of dispersed or precipitated particles could influence recovery and recrystallization behaviour of the alloys in the early days (97-110). Even though the results were not same, some of them showed particle could accelerate recrystallization (98,100,105) but some displayed retardation of the recrystallization by the particle (99,101,104,108), the effect of the particle is ineligible.

In a study of recrystallization behaviour of a rolled Ca-Mg-Sn-Pb alloy, Beck found that the alloy recrystallized within one day at 150°C when the alloy was quenched from 300°C previous to rolling, but when the quenched material was heat treated to the stage with maximum age hardening before it was rolled, it did not recrystallize for several
Fig. 1.3 Variation of the interparticle spacing (R in µm) against the time for 50% recrystallization. The open circles are the uniform crystals, the filled circles are for alloys artificially nucleated, and the dotted line gives the corresponding results for the polycrystalline alloys (103).
months at 150°C. When the material was overaged by annealing before rolling, the time of recrystallization was less than 6 hrs. at 150°C (97). Apparently, it is the state of dispersion of the precipitate before rolling that influences the time of recrystallization. This was one of the earliest reports about the effect of dispersed particles on recrystallization of the alloys. This study not only showed that the particles did affect recrystallization but also displayed that the effect depended on particle state.

A detailed study of particle effect on recrystallization was carried out by Doherty and Martin in aluminium-copper alloys in early of the 60's (102). The alloys containing different percentage of copper were heat treated at different temperatures to obtain different volume fraction and size of θ dispersions, and then were cold deformed followed by recrystallization annealing. They found that the matrix containing the coarser dispersions recrystallized faster than the single-phase solid solution. With finer dispersions, the recrystallization was increasingly retarded, becoming much slower than in the solid solution. Doherty and Martin compared their result with others', and found that the effect of the particle on recrystallization depends on interparticle spacing. They claimed that all reported cases of retardation of recrystallization have occurred in the field of metal-metal-oxide alloys, where the interparticle spacing has been of submicron size. Results of work in which acceleration has been observed showed interparticle spacing of 8-16 μm.

In another study, Doherty and Martin described the relationship between interparticle spacing (R) and recrystallization time \( t_{1/2} \) (the time for 50% recrystallization) in aluminium-copper alloys (103). The plot of R vs \( t_{1/2} \) is shown in Fig.1.3. It shows that an alloy having a mean interparticle spacing of 4μm recrystallized at a rate which was approximately 200 times faster than that of a solid-solution alloy having the same composition as the dispersion matrix. However, decreasing the spacing to
approximately 1.6μm retarded recrystallization by approximately 10 times, and a further reduction of the spacing to 1.2μm retarded the recrystallization process by a further 1000 times. It also shows that in alloys with a coarse dispersion, the rate of nucleation is fast and not very sensitive to changes in R while below a critical value of R (0.8 to 1μm), the rate of nucleation falls very rapidly with R. However, the acceleration of recrystallization with increasingly coarser dispersions can not continue indefinitely since there must be a return toward the value for the single phase alloy (103). This could be explained as that interparticle spacing (R) is not only factor of the effect on recrystallization. When the spacing increases to a certain value (or so called up limit value), its effect will vanish.

Mould and Coterill investigated the effect of particle content on the recrystallization in aluminium-iron alloys which contained dispersions distributed in the interparticle spacing larger than 4μm (105). It was found that, as the spacing increased, the acceleration effect decreased. For instance, the alloy containing dispersion with spacing of 4.5μm recrystallized at about 270°C after one hour, but the alloy with 15.4μm spaced dispersions fully recrystallized at about 340°C. It was also found that the alloy with 15.4μm spaced dispersions recrystallized at a rate about 10 times slower than that of the alloy with the 4.1μm spaced dispersions. However, further decrease in the interparticle spacing could restrict recrystallization. Mould and Cotterill explained particle effect on acceleration of recrystallization as that the introduction of a small number of uniformly dispersed hard particles to an alloy will provide additional nucleation sites at the particle/matrix interfaces, with a consequent acceleration of recrystallization. Further increases in particle density, decrease in interparticle spacing, will then lead to a continued increase in the nucleation rate. However, as the particle density increases, the interparticle spacing will be reduced to a critical value, at which no enough space for the nuclei being viable simultaneously at all the particles because
their embryos will interfere with each other before reaching a viable size. Hence, for particle density greater than that represented by this critical value, there will be a reduction in the overall nucleation rate leading to a second critical condition when the interparticle spacing is of the same order as the diameter of the cell structure in the deformed matrix. At this stage, the cell walls are likely to be pinned by the particles, and their coalescence to form viable nuclei will be entirely prevented. This can be seen from Doherty and Martin's results in which recrystallization was retarded by small spaced dispersions in the way of walls pinning (103).

From the results mentioned above, it can be drawn that presence of the particles will affect nucleation rate significantly. When the interparticle spacing is larger than nuclei size (cell or subgrain size), the particle will act as nucleation sites, then recrystallization is accelerated. If the spacing is smaller than nuclei, the particles will retard nucleation. Therefore, it is necessary to understand interaction between the dispersed particle and matrix structure during deformation and following heat treatment which will affect nucleation.

1-3-2 Effect of Dispersed Particles on Nucleation of Recrystallization

Since there are two kind of statements about effect of the particle, retard recrystallization and accelerate recrystallization, they will be reviewed separately. Firstly, we will discuss how the particle retard nucleation.

1-3-2-1 Retarded Nucleation in Dispersion Alloys

According to the theory of nucleation (106,107), nuclei form due to subgrain coalescence by slow migration and during growth the angle and mobility increase until
high angle, mobile boundaries form. As mentioned above, when the interparticle spacing is similar or smaller than cell structure or subgrain size, the cell walls are likely to be pinned by the particles, and cell coalescence to form viable nuclei will be entirely prevented (102,105). Particle pinning subgrain boundaries was considered as one of reasons that retardation of the nucleation in the dispersion strengthened alloys (102,103,105,111).

Nucleation in grain interiors preferably occurs on transition bands or other types of local misorientation which resulting from inhomogeneous deformation (112,113). Increase in degree of inhomogeneous will enhance nucleation rate. However, It was found that the presence of fine uniformly distributed dispersions could cause more homogeneous deformation in the alloys. Studies carried out by Brimhall et.al. (108), and Humphreys et al. (109,110) showed that retardation of nucleation in dispersion strengthened alloys is resulted from homogeneously deformed structure due to the presence of fine dispersions.

In the Brimhall's work, it was found that, in Ag-MgO alloy with fine dispersions, subgrains formed upon annealing were surrounded by relatively low angle boundaries resulting from the small magnitude of the misorientation in the cell structure. This could prohibit cell structure or subgrains to form nuclei. It was considered that the retardation could be attributed to the nature of the deformed structure, which did not contain regions of severe plastic curvature. As a result, the formation of mobile interfaces by misorientation accumulation was difficult (108).

Humphreys and Martin gave a more clear description by observation and analysis of interaction between the dispersions and dislocations in two phase copper crystals (109). They found that the precipitates tend to promote a much less inhomogeneous
structure on the slip plane since the distance is reduced between slip planes containing high densities of dislocations, then the lattice misorientations across these planes are reduced. This means that the presence of the dispersions could result a more homogeneous deformed structure than single phase alloy. Homogeneous structure will reduce nucleation rate, thus this was considered as another reason of retardation of nucleation by the dispersions (109,110).

In a study of low oxide Al-Al₂O₃ alloys, Hansen and Bay have considered another way in which dispersed particles may affect nucleation, that is, the movement of individual dislocation during recovery is limited owing to pinning by particles (114). This will retard recrystallization in two possible ways. Firstly, Individual dislocations are present within the subgrains after deformation and the particles may limit their movement to the sub-boundaries, thereby reducing the speed at which the sub-boundary misorientation increases during recovery. Another possibility is that subgrain growth during recovery involves the movement of individual dislocations from a sub-boundary to neighbouring boundaries, and this movement is limited by the particles.

Among the three possibilities of retardation of nucleation by the particle mentioned above, dislocation pinning and sub-boundary pinning have been observed in several studies lately in Al-Al₂O₃ alloys (111, 115-117). It was confirmed that subgrain coalescence is an important factor in the development of both inter- and intragranular nuclei. The oxide dispersions appear to impede this coalescence process and also to reduce the mobility of recrystallizing interfaces during the growth stage (111).

These possible ways which nucleation is retarded by the particles could occur simultaneously or individually in alloys containing fine and close dispersed particles.
Fig. 1.4 The orientation of the matrix in the vicinity of a 4μm diameter particle (113).
Whatever they occur, particle size and interparticle spacing are main factors which can change the behave of the particles in the alloys.

1-3-2-2 Nucleation at the Particles

As mentioned above, coarse or large spaced particles will accelerate nucleation because those particles could act as nucleation sites. Several studies have displayed electron transmission micrographs in which new grains recrystallized on the particles (113). Martin described nucleation on the particle as that the particle-matrix interface is a region where, on plastic deformation, there is likely to be high lattice curvature. Thus the dispersed particles may be regarded as producing an effect on recrystallization behaviour comparable to that of an array of grain boundaries (98). This was only a suppose which describes nucleation mechanism on the particle since lack of microstructural observation and analysis. The mechanism of nucleation on the particle was not understood very well until the deformed structure at interface and the orientation relationship between deformed and recrystallized structures around particle were studied.

In the work carried out by Humphreys (113), the orientation relationship between deformed and recrystallized materials around particles was studied using X-ray analysis and TEM observation in Al-Si-Cu alloy. It was found that deformation would produce deformation zones in the matrix near the particle-matrix interface. Those deformation zones could have up to 35° misorientation with the matrix around a particle with a diameter of 4μm. As the distance increases, misorientation decreases as shown in Fig.1.4. The in-situ annealing showed that those deformation zones acted as nucleation sites. The origin of a deformation zone at a particle is described by Humphreys as shown in Fig.1.5 (a-c). Consider the matrix containing a particle is
Fig. 1.5 The origin of a deformation zone at a particle (113).

Fig. 1.6 The annealing behaviour of a deformation zone (119).
Fig. 1.7 Semi-quantitative representation of the effect of strain and particle size on the deformation mechanisms at particles in aluminium (119).
deformed (Fig. 1.5a), several slip planes intersect the particle (Fig. 1.5b). If the particle is non-deformable then local plastic deformation must restore the particle to its original shape leaving the matrix sheared. This would produce a local reverse shear resulting in a series of inner subgrains with progressive misorientations (Fig. 1.5c). During annealing, recovery will occur eliminating many stored dislocations, and arranging the geometrically necessary dislocations into sub-boundaries within the deformation zone as shown in Fig. 1.6a. The nucleation process is similar to the process of polygonization, but it is much faster than the matrix outside of the zones because of higher dislocation density and smaller subgrains in the zones. Then the nucleus originating in the zones grows at an early stage towards and around the particle as shown in Fig. 1.6b and c. Finally, the zone will be consumed by growing of the nucleus as Fig. 1.6d. If the particles are located at grain boundaries or transition bands, nucleation will preferentially occur at those particles because nucleation within the deformation zone is favoured when such heterogeneities are present (118).

A further observation and analysis of deformation zone and local lattice rotation at particles was conducted by Humphreys (119, 120). It confirmed that for large particles (>0.1μm) and strains, the inhomogeneous plastic deformation at particles is accommodated by local lattice rotations. The rotation for a given strain decreases as the particle size is reduced. The lattice rotation depends on both strain and particle size as shown in Fig. 1.7. It can be seen that for small strain and particle, prismatic loops are formed as a mechanism of stress relaxation around the particles, but for large strain and particles, lattice rotation in the deformation zones will occur. The rotation depends on both strain and particle size, but for particles of diameter greater than 2μm, maximum rotation is independent of diameter, and increases linearly with shear strain (121).
Fig. 1.8 The conditions of deformation and particle size for which nucleation is observed to occur at particles (113).
Since nucleation at the particle depends on lattice rotation in the deformation zones, nucleation at particles will depend on strain and particle size as shown in Fig. 1.8. This figure was summarized by Humphreys after observation of nucleation at the Si particles in Al alloy (113). It was found that, in the area below the line with circles, nucleation occurred only at a minority of the particles, and above the line with filled circles, nucleation has occurred at most of the particles. It can be seen that nucleation at particles occurs or not depends on particle size. When the size is larger than 2\( \mu \)m (from Figure 1.8), the particles will act as nucleation sites. If the size smaller than this value, nucleation rarely occurs at the particle whatever deformation is how large. The critical size of the particle for stimulating nucleation decreases as deformation increases. The particle observed to stimulate nucleation could be as small as 1\( \mu \)m in Cu-Ag-Si alloy (122).

It should be mentioned that lattice rotation is a condition for nucleation, but vice versa is not true. It was found that rotation could be formed at particles of diameter of the order of 0.1\( \mu \)m, but nucleation does not occur at particles of diameter less than 1\( \mu \)m (113, 123). This is because existence of critical particle size. Only the particles larger than the critical size could provide sufficient driving force for the nuclei to grow. The critical particle diameter is the order of 1\( \mu \)m (113). However, even at large particles, nucleation could not always occurred. It was observed that many large particles were found without any nuclei nearby in 90% cold deformed aluminium of commercial purity after annealing at 300°C for 15 minutes (124). This could be due to either no lattice rotation occurs or it occurs but too small to generate nucleus.

From the analysis and microstructural observation mentioned above, effect of the particle on the recrystallization could be summarized as dependent of particle size and interparticle spacing (125). In the alloy containing fine closely spaced particles,
recrystallization will be retarded as results of sub-boundary pinning and homogeneous distribution of dislocations. If the fine particles are widely spaced, their effect will be limited. Coarse widely dispersed particles will stimulate nucleation, and accelerate recrystallization. There is evidence that nucleation occurs preferentially at pairs or groups of particles, even the individual particles are smaller than 1µm (115,126). However, nucleation in this kind of alloy with coarse closely spaced particles is not clear (125,126).

The recrystallization behaviour in the alloy containing both large and small particles was studied by several researchers (115, 118, 127-129). The recrystallization kinetics in the Al-Al₂O₃ alloy were retarded due to the presence of fine alumna particles and accelerated due to the presence of large FeAl₃ particles. The net result of having both types of particles might be small and could be a retardation or an acceleration (115). Romanowski and Cotterill compared recrystallization behaviour of alloy containing both large and fine particles with alloys containing either large particle or fine particle (128). It was found that the behaviour of alloy containing mixture of coarse and fine particles could be seen as a compromise between the separate behaviours of the single dispersion alloys. The recrystallization was initiated at the coarse particles but was gradually suppressed by the influence of the fine particles as the annealing temperature decreased. However, it was also found that even the large particles acted as nucleation sites, the overall kinetics of recrystallization was controlled by fine particles which had a severe retarding influence in nucleation (129).
Fig. 1.9 Apparent nucleation and growth rates versus inter-particle spacing for aluminium-copper alloys (the open circles - after Doherty and Martin) and for aluminium-iron alloys (the filled circles - Mould and Cotterill) (105).
1-3-3 Recrystallized Grain and Grain Growth in Particle Containing Alloys.

1-3-3-1 Effect of Particles on Grain Size

There are several concerns about nuclei growing to new recrystallized grains in alloy containing particles since the rate of growth of the nuclei is different in alloys containing large particles from fine particles. One concern is that if the particles act as nucleation sites, the rate of growth of the new grains will depends on the interparticle spacing as shown in Fig. 1.9. For alloys having interparticle spacing greater than $C_1$, viable nuclei will be formed at each particle. The migrating boundaries will impinge on each other before coming into contact with other particles. Therefore, the rate of growth will not be significantly influenced by particle density, and the grain size will decrease as the spacing decreases. When the spacing is less than value at $C_1$, mobile boundaries, originating at fewer nucleation sites, will have to move past those particles which have not produced an active nucleus before impinging on other migrating boundaries. In this case, growing rate is reduced. Slow growth of a small number of nuclei will generate a less uniformly distributed coarse grain structure (105).

Several researches have shown that the grain size is dependent on interparticle spacing (98,102,103). It was suggested that the equivalent grain size related to particle spacing as $G=4r/3f$, $G$ is net radius of curvature of grain, $4r/3f$ defined as interparticle spacing in which $r$ is radius of the particle, and $f$ is volume fraction (98). It was also supposed that if each particle was to nucleate one grain, then the grain size of alloy may be determined directly by the interparticle spacing (113,120,124).
Some studies have shown that recrystallized grain size is actually controlled by nucleation rate rather than growth of the nuclei (102,105). High nucleation rate or an acceleration of recrystallization will yield a finer grain size, and retarded nucleation will produce large grain (102,103,113). Then control of the nucleation rate by dispersed particles in alloys could be used to control the grain size (124,130).

Second concern is that presence of fine dispersions will retard recrystallization, and its effect on grain size. There could be two kinds of responses of recrystallized grain on the effect. One is that grain size increases as recrystallization is retarded (103). It was considered that particle-retarded recrystallization should be typified by a less uniformly distributed, coarse grain structure resulting from the slow growth of a small number of nuclei (105). However, if those fine dispersions could inhibit the growth of all the potential nuclei, developing grains can only grow slowly. The incubation of nucleation enables more grain to nucleate, and subsequently, fine recrystallized grains can be generated. (131).

Finally, in alloys containing both large and fine particles, recrystallized grain size and distribution will depend on proportion of large and fine particles. As the volume fraction of large particle increases or fine particle decreases, final grain size will decrease (124, 129, 132). It was found that growth of the nuclei at large particles could be restrained severally by fine particles resulting in a large number of small "island" grains surrounding large particles (127, 129). It seems that nuclei at the particles only assume deformation zones, then further growth is restricted by fine particle pinning resulting in a mixed structure.

Nes developed a model to predict recrystallized grain in alloy containing bimodal particles (132). It was found that grain size increased as fine dispersion level f/r
increased. This was also observed in Chan and Humphreys' work, even quantitative agreement was poor (129). It was considered that, with same amount of large particle, nucleation events decreased as fine particles increased resulting in large final grains. However, if fine particles further increased, the pinning became sufficient to prevent growth of any nuclei, then final grain size would decrease (131).

Nevertheless, recrystallized grain size is affected by particle size and distribution. Relation between grain size \( R \), particle size \( r \), and particle volume \( f \) was first described by Zener as \( R = \frac{4r}{3f} \) (133). This relation was derived from a balance of driving force for grain growth and restraining force from the particle. It was also used to describe the growth of subgrains during recrystallization (134). Even though this model was found not accurate in some case because the driving force for grain growth was found to be overestimated in this model (135), it was considered as a basic description of the relation between grain size and particle state (134).

Another model developed by Humphreys has related the recrystallized grain size \( D \), in the case of particle stimulated nucleation, to the particle size \( d \) and volume fraction of particle \( f \) randomly distributed throughout the matrix (136). Then the grain size following recrystallization can be determined to be:

\[
D = \frac{d}{f^{2/3}(1-f)}
\]

This model will give an upper limit grain size because the particle larger than 3\( \mu \)m may nucleate more than one grain, and then final grain size will be much finer (136,137).
1-3-3-2 Grain Growth in Alloy Containing Particles

Grain growth in alloy containing particles could be affected by the pinning of a grain boundary migrating through a dispersion of particles. This could be described by well known model developed by Zener (133). The limiting size $D_{\text{max}}$ to which grain grows, in the absence of abnormal grain growth may be taken as:

$$D_{\text{max}} = \frac{2d}{3f}$$

Several studies show that this model often overestimates the driving force and gives much larger grain size than that observed (135, 138,139). Even though this model has been shown not accurate because of ideal conditions supposed during derivation, it still represents a useful approach to the estimation of the limiting ultimate grain size in dispersion alloys, and is used broadly up to now (134,136,137). This is because that this model has simply described the relation between the grain size and the particle parameters such as particle size and volume fraction.

The other models have been developed with different approaches such as Gladman's model based on the energy changes accompanying grain boundary unpinning process (135, 140). It was found that grain growth after recrystallization could occur when the particles coarsened to a size in excess of the critical radius. The critical size of the particle was derived from balance of the maximum rate of increase in energy involved in the unpinning process, and the rate of change of energy due to grain growth. It was related to particle volume fraction, the matrix grain size, and the heterogeneity of the structure (135).
Hillert's model based on grain-size distribution theory suggested that normal grain growth should stop when the critical grain size $R_{cr}$ reached to $1/3z$, $(z=3f/4r)$, i.e., $D_{cr}=2/3(4r/3f)$ (141). It can be seen that this value is smaller than that predicted by Zener's model.

Ashby's analysis based on the soap film model allows the pinning force exerted by a single particle on a boundary to be measured, and get similar prediction of grain growth as Zener's estimate (142,143).

Even other theoretical treatments employ different approach to describe grain growth in particle containing alloys, they display almost similar conclusion that the limiting ultimate grain size increases as the particle size increases and/or as the particle volume fraction decreases similar to the description in equation 1-3-2 (134).

1-3-4 Recrystallization Behaviour in Discontinuous Metal Matrix Composites

Study of the recrystallization behaviour in particle or short fiber reinforced metal matrix composites has been carried out in recent years as the attention has been paid to thermomechanical processes of the composites (136, 137, 144-149). Studies show that recrystallization behaviour of the composites is similar to that of the alloys containing large particles mentioned in former sections. The kinetics and recrystallized grain size may be broadly interpreted in terms of models developed for conventional two-phase alloys (136). However, comparing to conventional two-phase alloys, the composites contain the particles much larger in size and much higher in volume fraction. Distribution of the particles or whiskers could be less uniform, and close spaced whisker or particle groups were often found in the materials (144).
Furthermore, most of the composites containing relatively high volume fraction of fine dispersion and inclusion (137, 144-146). All these factors in the composite could give it a different recrystallization behaviour from that of the dispersed alloy.

A series of detail investigations of deformed structure, annealed starting structure, and recrystallized structure in aluminium alloys containing up to 35% volume fraction of SiC whiskers or particles were carried out by Humphreys (136,148), Liu, Hansen, and Jensen (144, 146,147). Several observations can be made.

1. The subgrain size decreased after adding SiC whiskers. The total dislocation density was higher compared to the matrix alloy for the same degree of cold deformation (144). This is attributed to a higher concentration of geometrically necessary dislocation caused by the presence of hard SiC fiber (50). The main features of the dislocation structure are large lattice rotations adjacent to the particles (136).

2. Dynamic recovery was observed in the composite which showed the sharpening of dislocation walls and subgrain boundaries. It was also found that subgrain growth in the composite was faster during the initial stage of annealing (146).

3. Nucleation firstly occurred at large particles or fiber groups. The number density of nuclei was higher in the composite than in matrix alloy. The temperature of nucleation decreased with the adding of SiC particles or whiskers. As the volume fraction of reinforcement increased, recrystallization rate increased. However, when the volume fraction was so high that interparticle spacing was smaller than certain value, the reinforcement would retard recrystallization (136).
4. It was found that the concentration of SiC groups which may be potential nucleation sites is much larger than the concentration of nuclei which grow into recrystallized grains (144). This was considered due to several reasons. Firstly, the strong pinning effect of the fine dispersion on the structure considerably reduces the capacity of SiC fibers to stimulate nucleation. Secondly, the limited volume enclosed by fibers is probably not suitable to produce a nucleus which can continue to grow. Thirdly, at high levels of deformation, dynamic recovery may reduce the number of potential nucleation sites at whiskers. Therefore, it was considered that only those which were associated with high-angle boundaries or transition bands may stimulate nucleation (118,144).

5. Recrystallization kinetics could be accelerated by a high nucleation rate in the composite. However, if content of fine dispersions is high, the kinetics will be retarded. The net result depends on parameters such as the size and spacing of particles (144,146).

6. Introduction of SiC reinforcement has a grain refinement effect due to a higher nuclei density and lower growth rate. This effect appears to be related to the pinning of SiC to high angle boundaries and the impingement of the nuclei of high number density (136, 144, 146). Recrystallized grain size increases as particle size increases in the alloys containing same volume fraction of reinforcements (149). For instance, average grain size increases from about 12\(\mu\)m to 170\(\mu\)m as the particle size increases from 7\(\mu\)m to 100\(\mu\)m in alloys both containing 30% volume fraction of reinforcement. This is because that the interparticle spacing is much larger in larger particle composite. Therefore, particle spacing is very important factor for recrystallized grain size.
Those observations have also been made in other reinforced materials (136,137,146-149). The results of the observations appear to be similar but some other aspects are investigated. For instance, it was found that the nucleation efficiency per large particle increased sharply with increasing volume fraction of the reinforcement. Nucleation occurred at only about 10% of the reinforcement particles in alloy containing 10% volume fraction of SiC particles. As the volume fraction increased to 20%, nucleation occurred at about 30% of the particles (145). This was considered due to an increased plastic misfit strain in alloy with high volume fraction of the particles. However, it requires a further study in the deformation structure and the annealing structure.

Grain growth was found to be retarded in reinforced materials by both fine dispersions and reinforcement (145). This can be seen from the fact that most of the particles are located at the grain boundaries, and only few particles become engulfed by grains. It supposes that interparticle spacing restrict grain growth in the materials. The limiting grain size to which grain grows in the composites may be estimated by equation 1-3-2 (136).
1 - 4 Deformation Behaviour of the Composite at Elevated Temperature

One of prime advantages of the aluminium MMCs is that the billets of the composites can be deformed to needed shape and size by conventional secondary fabrication processes such as extrusion and rolling. Like most metallic materials, these deformation processes are generally conducted at elevated temperatures and thus it is important to understand deformation behaviour and microstructural development of the composite during deformation at high temperatures.

Since deformation behaviour of the composites can be studies by conventional theories such as analysis of flow stress curves and restoration processes. It is necessary to review briefly hot working theories of the metals with the approaches of mechanical behaviour and microstructural changes during deformation.

1-4-1 Dynamic Restoration Processes of Metals During Deformation

Recovery and recrystallization have traditionally been considered as mechanisms of restoration through which cold-worked metal returns partially or completely to its condition prior to working. 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, recovery and recrystallization can also take place under stress and during concurrent straining, i.e., under conditions of high temperature deformation. In such an event, they are distinguished from the respective static processes by the terms of dynamic recovery (DRV) and dynamic recrystallization (DRX) (150). These two processes are considered as main restoration processes during hot working of the metals. Dynamic recovery and recrystallization are well distinguished by their
Fig. 1.10 Stress strain curves of several materials, DRV alone in Al and Al-1Mg alloy, and DRX in Al-5Mg, Al-5Mg-0.7Mn, Cu and Ni alloy (151).
microstructural development during deformation. Dynamic recovery is characterised by substructure with low angle boundaries within elongated previous grains. On the other hand, dynamic recrystallization will generate new equiaxed grains with large angle boundaries during deformation. These two processes can also be identified by the stress-strain flow curves. Typical stress strain curves of several materials under approximately equivalent conditions are shown in Fig.1.10. It can be seen that Al, and Al-1.0Mg alloys display a different type of curves from those of Al-4.5Mg-0.7Mn, Cu, Fe-18.3Cr-8.7Ni, and Ni alloys. This is because they undergo different restoration mechanisms, or called softening mechanisms (151). The former two alloys have shown a typical curve of dynamic recovery, and later four alloys have displayed a typical curve of dynamic recrystallization. These two softening processes will be reviewed separately in the following.

1-4-1-1 Dynamic Recovery

1-4-1-1-1 The Flow Stress

The typical shape of the flow curve for restoration by dynamic recovery is shown by Al and Al-1Mg alloys in Fig. 1.10. During the initial straining, dislocations are generated and they become entangled, thus raising the flow stress (152). As the deformation continuing, the dislocations annihilate and rearrange into neatly arrayed low energy walls which define polygonized subgrains then the rate of work hardening decreases. Finally, when the rate of dislocation annihilation equals the generation rate then an equilibrium dislocation density is maintained despite increasing strain. The constant dislocation density results in a steady state stress. The steady-state stress level depends on deformation temperature and strain rate. A general relationship between flow stress and strain rate, at constant temperature, is
\[ \sigma = K \dot{\varepsilon}^m \]  

where \( m \) is the strain rate sensitivity, and it can be obtained from the slope of a plot of \( \ln \sigma \) vs \( \ln \dot{\varepsilon} \). The \( m \) value increases as temperature increases, and it indicates changes in deformation behaviour with deformation temperatures (153). The relationship between the stress and the strain rate in a high stress case can be written as:

\[ \dot{\varepsilon} = A' \exp(\beta \sigma) \]  

and for all ranges of stress, it can be defined by the single relation

\[ \dot{\varepsilon} = A'' (\sinh \alpha \sigma)^{n'} \]  

where \( A', A'', \beta, \alpha \) and \( n' \) are constants (154).

If considered effect of the temperature, the relationship for correlating stress and strain can be described by:

\[ \dot{\varepsilon} = A_1 (\sinh \alpha \sigma)^{n'} \exp\left(\frac{Q}{RT}\right) \]  

where \( A_1, \alpha, \) and \( n' \) are experimentally determined constants and \( Q \) is an activation energy, \( R \) is the gas constant, and \( T \) is deformation temperature (155). However, in some cases, the relationship can be expressed by:

\[ \dot{\varepsilon} = A_2 \exp(\beta \sigma) \exp\left(\frac{Q}{RT}\right) \]
where $A_2$ and $\beta$ are constant (156). It is mostly fitting the high stress cases (154,156).

For low stress case, the relationship between the stress and strain rate at different temperatures can be written to:

$$\dot{\varepsilon} = A_3 \sigma^n \exp\left(\frac{Q}{RT}\right)$$  \hspace{1cm} 1-4-6

where $A_3$ and $n$ are constants.

The equations 1-4-4–6 are also used to evaluate activation energy for deformation, $Q$, by plotting $\ln \sigma$ vs $\ln \dot{\varepsilon}$ (150,156-158). If the $Q$ values are known, the correlation of temperature and strain rate to the flow stress (peak stress or steady-state stress) can be described by the following equation:

$$Z = \dot{\varepsilon} \exp(Q/RT) = A_1 (\sinh \alpha \sigma)^n$$  \hspace{1cm} 1-4-7

where $Z$ is the temperature compensated strain rate (Zener Hollomon parameter) (159-163). It can be seen that the flow stress depends on strain rate and temperature according to this equation.

1-4-1-1-2 Microstructural Development During Dynamic Recovery

The main features of the substructure, such as the dislocation density between the walls, the average spacing between them and the misorientation across them do not change during the course of steady state deformation (164). This means that the substructures by this time only depend on the alloy, and on the strain rate and temperature of deformation but not on the strain (150). Figures 1.11(a-c) show the substructures produced in commercial purity aluminium by extrusion at a strain rate of
Fig. 1.11 Hot deformation structure in commercial purity aluminium by extrusion at a mean strain rate of 1 s⁻¹, a) 25°C, b) 350°C, and c) 450°C (150).
At low temperature, deformed material displayed the cell structure with high density of dislocation. As deformation temperature increased, subgrains became coarse.

The mechanisms for dynamic recovery are usually described as the thermally activated processes of dislocation glide, climb and cross-slip (152,165). Therefore, any factors which affect dislocation movement may affect the level of recovery. Beside deformation temperature and strain rate, those factor, including the stacking fault energy (SFE), solute atoms, and second phase may influence recovered substructures (166). The stacking fault energy has been considered as one of most important factors that affect recovery of the alloys since it controls the ability of dislocations to cross-slip or climb (150). The metals such as silver, gold, and copper have low SFE, and aluminium and iron have high SFE (166,167). It was found that substructure in low SFE metals displays dissociated dislocations in irregular forests, and no clear cell structures could be formed (168,169). However, a clearly defined strain-free cell structure is usually found in high SFE metals as shown in Fig.1.11. It should be mentioned that the stacking fault energy also affects the dislocation density of a deformed metal and recrystallization stored energy (170,171). Generally, the dislocation density of deformed metal increases as the stacking fault energy decreases, and the stored energy for recrystallization also increases as the stacking-fault energy decreases. Thus metals with low stacking-fault energy often undergo dynamic recrystallization whereas metals with high SFE generally possess dynamic recovered rather than recrystallized structures during hot deformation.

Solute may reduce recovery either by diminishing the SFE or by providing atmospheres that reduce dislocation mobility and possibility of climb by binding vacancies (150,151,166). Several investigations in different metals show that
Fig. 1.12 The subgrain diameter increases as the temperature-compensated strain rate $Z$ decreases (150).

Fig. 1.13 Two types of flow stress-strain curves that show dynamic recrystallization during hot working of the alloys (150).
increasing the solute content leads to a decrease in stacking-fault energy which in turn influences the recovered structures (172,173).

Particles reduce recovery by pinning dislocations in the walls and by locking the subgrain boundary nodes (174-176). The effect of particles on cold worked structure has been reviewed in former section. The same effect could occur during hot deformation, that is, widely-spaced large particles promote the formation of a cell structure since they act as dislocation sources whereas only closely-spaced fine particles reduce recovery by pinning dislocations (177). The effect of particles on restoration processes will be discussed in detail later.

The recovered structures also depend on deformation temperature and strain rate. The subgrain size $d_s$ is related to the temperature compensated strain rate $Z$ by the equation:

$$d_s = a + b \log Z \quad \text{or}$$

$$d_s^{-1} = a_i + b_i \log Z \quad \text{1-4-8}$$

where $a$, $a_i$, $b$ and $b_i$ are empirical constants, and $Z = \dot{\varepsilon} \exp(Q/RT)$ (150,151,160-162). Figure 1.12 shows experimental result which correlates subgrain diameter to logZ in a linear relationship described in equation 1-4-8. It can be seen that the subgrain size increases as the temperature compensated strain rate decreases (i.e., temperature increases). The data fall into a narrow band between two dash lines indicating that there are compensating decreases in diameter associated with the increases in activation energy (150).
1-4-1-2 Dynamic Recrystallization

The primary softening mechanism is dynamic recovery which counteracts the strain hardening of dislocation interactions, starting from the point of yielding up to a potential saturation regime in which annihilation and generation rates balance. However, if the dislocation substructure continues to rise in density, it eventually creates nuclei and provides driving force for dynamic recrystallization (150,178,179). This is often observed in alloys with low stacking-fault energy such as Cu, Pb, Ni, and γ-Fe in which the rate of dynamic recovery is limited (151). The flow stress curves and microstructure in the metals undergone dynamic recrystallization are different from that of dynamic recovery since dynamic recrystallization increases the rate of softening by completely eliminating the substructure as the grain boundaries migrate. The result is a peak and work softening to a steady-state regime (151). Dynamic recovery is essentially a re-arrangement of dislocations whereas dynamic recrystallization involves the annihilation of dislocations by a sweeping high angle boundary (180). Dynamic recrystallization could be characterized by flow curves and microstructure;

1-4-1-2-1 The Flow Curves

The schematic flow stress curves during dynamic recrystallization are shown in Fig.1.13. It shows that hot working at a low strain rate leads to a cyclic stress-strain curve caused by waves of recrystallization. At high strain rates, recrystallization starts at higher strains, causing the drop in stress after the peak. When the strain rate is low, recrystallization proceeds to completion. Once it is complete, the dislocation density builds up again, then the flow stress rises until recrystallization is again nucleated. This repeated process gives the flow curves a wave shape (150,183, 184). In the high
strain rate deformation, the dislocation density within first recrystallized grain has increased sufficiently that second nucleation occurs before first round of recrystallization is complete. Thus, at any instant, there is a distribution of regions with different degree of deformation and dynamic recrystallization. Reaction of these two processes results in a steady-state flow stress after softening from peak value (150).

1-4-1-2-2 Recrystallized Grains

Dynamic recrystallization is a discontinuous process which includes nucleation as a result of misorientation build up between subgrains and growth of the nuclei (181,182). Nucleation occurs most frequently at existing grain boundaries but is also found at twin boundaries and deformation bands while strain rate is high (179,180,183,185,186). The new grains grow as necklaces along the original boundaries. Once the potent grain boundary sites are exhausted, the nucleation will continue at the interface between recrystallized and unrecrystallized grains (151,179,180). The size of these new grains $D_s$ is related to temperature compensated strain rate $Z$ by:

$$D_s^{-1} = a' + b' \log Z \quad 1-4-10$$

and relationship between steady-state flow stress and grain size is described as:

$$\sigma = c' + e' D_s^{-p} \quad 1-4-11$$

where $a'$, $b'$, $c'$, $e'$ and $p$ are constants, and $Z=\dot{\varepsilon}\exp(Q/RT)$ (151, 179, 186,187). It can be seen that dynamic recrystallized grain size is only dependent on deformation
conditions such as deformation temperature, strain rate, and activation energy of deformation. It is considered that, throughout steady state, the grain remains constant in size and almost equiaxed (150,151).

1-4-1-2-3 Effect of Alloying on Dynamic Recrystallization

Dynamic recrystallization often occurs in the alloys with low stacking fault energy, such as copper and γ-Fe. This is because mobility of dislocations is poor in those alloys. As deformation increases, high density of dislocations is easily built up, and nucleation of recrystallization is promoted (150). In alloys with high stacking fault energy, such as aluminium, cross-slip of dislocation is easy so that softening mechanism is controlled by dynamic recovery, and generally, dynamic recrystallization dose not occur (178). However, it has been found that dynamic recrystallization did occur in aluminium alloys when Mg or Zn were added (188). It was considered that addition of Mg or Zn could lower the stacking fault energy sufficiently for dynamic recrystallization. Even though other researches have shown different results in which only dynamic recovery was operating in these alloys, the addition of solute has been found having a significant influence on dynamic recrystallization (150). As mentioned in former section, solute could lower stacking fault energy and also could hinder the migration of boundaries then slowing the rate of dynamic recrystallization. This has been found in copper alloys by adding Ni or Al (150). In next section, effect of alloying on dynamic restoration of aluminium alloys will be discussed in detail.

1-4-2 Dynamic Restoration of Aluminium Alloys

Aluminium is a typical high SFE metal (150,167). Numerous investigations of the hot deformation of pure aluminium and dilute aluminium alloys have demonstrated that
Dynamic recovery occurs very easily and have generally stated that dynamic recrystallization does not occur (151,178,189,190). Dynamic recovery is considered to be only restoration mechanism in aluminium alloys during hot working. Therefore, all characteristics of dynamic recovery and relevant equations mentioned above can be applied to aluminium alloys. However, there are some alternative and moderation in restoration behaviour, besides of dynamic recovery, of aluminium alloys containing certain amount of alloy elements such as Mg and Zn. It was found that dynamic recrystallization occurred in these alloys (159,188,192-194). In the work conducted by Gardner and Grimes, Al-Mg alloys displayed a type of DRX curves and structures when Mg content increased to 5% (188). Dynamic recrystallization was also found in hot rolled and extruded Al-Mg, Al-Mn-Mg, and Al-Si alloys (193,194). Even in 99.99% pure aluminium, DRX could be found during hot rolling (194,195).

There are several reasons for DRX occurring in aluminium alloys. Firstly, it was considered that adding Mg or Zn into pure aluminium could lower stacking fault energy of the alloys, leading to reduce the static recrystallization temperature of cold worked alloys and enhance the possibility of dynamic recrystallization of the alloys (188,192). It was suggested that the addition of 1% Mg reduces the stacking fault energy from about 200 to about 50 mJ/m² (188, 196). Secondly, the solute drag could increase dislocation density in the substructure to initiate DRX during hot working (150,160). This was confirmed in several studies which showed that only dynamic recovery was operating in alloys containing up to 5% Mg at considerably high strain rate (160,189,190,197) but DRX occurred in the low strain rate testing (151,160). It was concluded that alternative in dynamic restoration behaviour in aluminium containing Mg or other alloys is caused by the solute drag instead of low SFE (160). In high strain rate case, dislocation velocities are so high that the solute drag is left behind, leading to low dislocation densities that can not initiate dynamic
recrystallization. At low strain rate, dislocation movement is retarded by high solute drag, leading to high dislocation densities that could initiate dynamic recrystallization (151,160). High strains can also cause dynamic recrystallization occurring. It was found that when extrusion ratios exceed to 500, dynamic recrystallization process will be activated in aluminium of 99.5% purity (198). Another reason for dynamic recrystallization occurring in hot deformed aluminium alloys is presence of second phase particles (151, 159,160,174,193-195,199). High density dislocations and lattice rotation, or called deformation zones generated by large particles could increase possibilities of dynamic recrystallization in the matrix adjacent to the particles. This will be discussed in more detail in next section.

However, there are several possibilities to misinterpret the deformed structures. For instance, in highly strained alloys, small grains with large angle boundaries could be formed by pinch off of elongated original grains. These new grains are not dynamically recrystallized from conventional recrystallization process, i.e., nucleation and growth, but are formed by a process called geometrical recrystallization (200,201). McQueen and co-workers considered that heavily deformed grains develop serrations and become pinched off when average thickness of grains falls below twice the subgrain diameter. The serrations in the enlarged perforation appear to be several new grains with large angle boundaries (151, 200,201). This proposition has been confirmed by several studies, and has been used to interpret phenomenon of so called dynamic recrystallization in some type of highly strained aluminium alloys (202-204).

The alloys undergone dynamic recrystallization during hot deformation normally display flow curves with sharp peak. However, this kind of flow curves can also be given by the solution treated aluminium alloys. In the work carried out by McQueen,
the effect of solutes and precipitate on hot working behaviour of several aluminium alloys was studied. It was found that the solution treated alloys exhibited flow curves with sharp peaks and considerable softening which was greater at lower temperature, and there was no approach toward a steady state (205). Declined flow curves were contributed to dynamic precipitation and the softening to coalescence along with dynamic recovery instead of dynamic recrystallization (205).

Another possibility for misleading interpretations of restoration behaviour in hot deformed aluminium alloys is malfunction of experimental. Static recrystallization could occur at high temperature very quickly, and if the samples are not quenched immediately after hot deformation, static recrystallization may occur. This is more likely occurring in experimental of industrial scale such as hot rolling or extrusion. It was therefore recommended that the correct interpretation of microstructures with respect to DVR or DRX in aluminium alloys (as well as other metals) requires both large field observations of the grain structure by optical microscopy and observations of the dislocation structure by TEM microscopy (152).

1-4-3 Effect of Particles on Dynamic Restoration Process of Aluminium Alloys

The presence of second phase particles can considerably influence static restoration process of aluminium alloys by either retarding or enhancing recrystallization. The influence of particles during hot working is also considerably scientific interest since the particles can simultaneously influence the stress-driven dislocation displacements, the degree of dynamic recovery and the progress of dynamic recrystallization (174). The particles discussed in here are non-deformable and temperature stable dispersions
or inclusions. Effect of dynamic precipitation and dissolution during hot deformation are not concerned.

1-4-3-1 Effect of Particles on Dynamic Recovered Structure

Effect of particles on recovered structure is caused by interaction between particles and dislocations and cell walls during hot deformation (152,159,162,199,201,207,208). Like effect of particles on static recovery in cold worked alloys, effect of particles on dynamic recovery also depends on particle size and distribution. Fine close-spaced dispersions strongly pin dislocation and sub-boundaries and produce fine subgrains (159, 162, 206).

Several studies have shown that fine particles generally stabilize the substructure and frequently determine its dimension (150,152,174,207). However, interaction between particles and sub-boundaries can be altered as deformation temperature increases. It has been found that, at 200°C, the cells in particle containing alloy are smaller by 20-40% compared to monolithic aluminium, but at 400-500°C, the subgrains in both are comparable in size (208). Microstructure shows that, at low temperature, the particles are largely in the cell walls, but as the temperature increases, more and more particles are inside the subgrains. Therefore, it is considered that the change in substructure size at high temperature is because the dislocations become able to climb or cross glide around the particles, pinning effect has been diminished (201). This is considered as a main reason that strength decrease as deformation temperature increases in dispersion strengthened alloys (201, 208).

The transition in dislocation-particle interaction behaviour as the temperature change (also as strain rate change) has been discussed by Humphreys and co-workers in detail
It was concerned that the behaviour of particle containing aluminium alloys at high temperatures may be divided into two regimes, one in which the dislocations interact with the particles, leading to the formation of extra dislocations, and one in which the particles do not significantly affect the microstructure (209). Even though this transition theory is mainly used to describe microstructural development around large particles at high temperatures, it could give a reason that fine particles normally lose ability to stabilize substructure at high temperature.

The recovered structure could be affected by fine dispersed particles, but normally not by large particles because large particles are less efficient to pinning dislocations or cell boundaries. However, the presence of the large particles could modify restoration process of the alloys by producing dynamic recrystallization.

1-4-3-2 Effect of Particle on Dynamic Recrystallization

Effect of particles on dynamic recrystallization may be concerned from two aspects. One is from interaction between dislocation structure and fine particles, and one from effect of large particles. Fine particles are possible almost completely to inhibit static recrystallization after cold working. However, the presence of fine second phase particles can give rise to dynamic recrystallization in aluminium alloys (160, 190). This seems to be due to the raising of the dislocation density as a result of dislocation being pinned by the fine particles (160, 174, 193, 190, 207). It has been found that recrystallization nuclei could form when a large number of dislocations cluster at small submicron size particles (193). However, some studies showed that the presence of fine particles could retard dynamic recrystallization by pinning subgrain and grain boundaries (199, 211). Those results are not contradictory since those studies use alloys containing different solute. Dynamic recrystallization has been found in Al-
$5\text{Mg-1Mn alloy (159,174,195), but not been found in Al-1Mg-0.8Mn alloy (199), both alloys contain fine particles. It seems that high alloying level enhances pinning efficiency of dislocation, and increases possibility of dynamic recrystallization. McQueen and Jonas have found that only when concentrations of 5-7\% Mg are present, second phase particles tend to give rise to dynamic recrystallization (208).}$

The presence of large particles (>0.6\mu m) can increase dynamic recrystallization in aluminium alloys (150,151,159,160,193,199,207,212). This is because large particles act as nucleation sites during hot deformation as described in static restoration process. Nuclei around large particles have been found in hot deformed Al-Mg alloys, and recrystallized grains are found mostly adjacent to large particles (159,160,193,195,213). However, particle stimulated dynamic recrystallization mostly occurs in alloys with high solute content. In pure aluminium or low solution aluminium alloys, dynamic recrystallization could not be found even large particles are present (151). Therefore, it can be concluded that effect of the particles on dynamic recrystallization of aluminium alloys is mostly dependent on the solute level. Whatever the solute can lower stacking fault energy or increases drag effect, it may hinder climb and cross glide of dislocations, increase pinning effect of fine particles and enhance lattice rotation around large particles, leading to dynamic recrystallization.

1-4-3-3 Effect of Dispersoid Particles on Deformation Behaviour

Dispersoid particles generally increase the rate of strain hardening and strength of alloys by dislocation pinning. However, as deformation temperature increases, strength of the alloys could drop sharply due to dislocation by passing, or to particle coarsening or deformation (201,214,215,216). Therefore, the alloys containing
dispersoid particles exhibit a high stress exponent and a high apparent activation energy for deformation (Q value) (159,201,215,217).

Activation energy for self-diffusion in pure aluminium is about 140 kJ/mole (218), and for hot deformation is 155 kJ/mole (219). However, activation energy for hot deformation in aluminium alloys containing dispersoid particles could reach to as high as three or four times that for self-diffusion in aluminium (217). High activation energy has been considered as one of important characteristics of deformation behaviour in dispersoid alloys, which is related to degradation of mechanical properties of the alloys at high temperature (201,217).

1-4-4 Deformation Behaviour and Dynamic Restoration in Discontinuous Aluminium Composites

Deformation behaviour at high temperature and microstructural development of particle or short fibre reinforced aluminium MMCs have attracted attention of researchers in recent years as researches on MMCs increase. However, much less information on hot working of Al MMCs is available compared to monolithic aluminium alloys, especially on microstructural development during hot deformation. Even so, differences in mechanical and deformation behaviour in aluminium MMCs will be discussed in the following using limited literature sources.

1-4-4-1 Mechanical Behaviour of Aluminium MMCs at Elevated Temperature

Discontinuous aluminium matrix composites are generally difficult to hot work due to their high flow stresses and low ductility (220-224). However, they can be extruded,
forged, rolled, or pressed into a variety of shapes if the processing temperatures and strain rates are chosen carefully (222-224).

Several experiments have been carried out by torsion, and compression using 6061 and 7090 aluminium alloys containing 10 to 20 vol% of SiC particles or whiskers (220,221,224). Some observations can be made from those studies. Firstly, the true stress-true strain curves for the aluminium MMCs are typical of the form expected when dynamic recovery occurs; that is, the flow stress rises sharply to a maximum and then approaches to steady-state value (221,225). Secondly, the studies show that the composites are much stronger than the monolithic alloys at low temperature, but approximately similar above 550°C (220,224). The flow stresses for both reinforced and unreinforced alloys declined monotonically as the temperature increase, but decreasing rate for the composites are higher resulting in similar deformation resistant for the two kinds of the materials. Finally, the flow stresses increase as strain rate rises in both the composites and matrix alloys at identical temperature, but increasing rate is lower for the composites. This means that the composites display a lower strain rate sensitivity 'm' or higher stress exponent 'n' compared to monolithic alloys. Since the high changing rate of the stresses with the temperature, addition of the smaller m values, the reinforced alloys display a much higher activation energy for deformation by comparison with the monolithic alloys. This mechanical behaviour has been found in several aluminium composites during hot torsion and compression as well as creep testing (221,224, 226-228).

The activation energy for deformation Q can be evaluated by equations 1-4-4 to 1-4-6. Creep law equation 1-4-6, which is considered suitable for low stress case, gives a nonlinear fit of the data, and gives continuously changed Q values within the temperature ranges studied in SiC reinforced aluminium composites (220). Equation 1-
4-4, which is suitable for whole range of stress level, gives a good linear fit to data for SiC reinforced 6061 and 7090 aluminium composites, and a single Q value for whole temperature range tested can be obtained (224).

No matter which equation was used, high stress exponent and activation energy exhibited by the composites have been considered as behaviour of dispersion strengthened alloys (DSAs) (220,224-227). However, the composites are different from dispersion alloys because reinforcements are much larger than dispersion, and they are non-deformable. It is obviously that the loss in strength at high temperature can not be due to dislocation by passing or to particle deformation which is suggested occurring in DSAs (201). Therefore, the reasons of high activation energy for DSAs may not be suitable for explanation of the same behaviour in the composites. However, there is a similar theory for description of microstructural development in the composites during hot working. It is the transition theory described by Humphreys based on relaxation theories and dislocation climb theories (209,210).

It is considered that strengthening of the discontinuous MMCs is contributed to microstructures in the matrix which contains high dislocation density and fine substructures introduced by the presence of the large reinforcements (8,24,32). These microstructures are quite stable at low temperature, and then give the composites high strength. However, as the temperature rises, mobility of the dislocations increases and climb and cross glide of the dislocations are easier resulting in fewer dislocations accumulated at the particles, and then strengthening becomes less sufficient. When the temperatures and strain rates reach to their critical values, all the dislocations arrived at particles will be removed by climb or glide, then microstructure of the composites will be not much different from that of the monolithic alloys (209,210). If this transition theory may be applied, it can give a reason that the composites display a similar
strength as the monolithic alloys at high temperature, and high activation energy for deformation.

1-4-4-2 Microstructural Development in Aluminium MMCs During Hot Working

Several studies have shown that aluminium MMCs generally display dynamic recovery during hot working (209,210,221,225,229). Even several workers have suggested that new grains may nucleate at large second phase particles in aluminium alloys, dynamic recrystallization has not been found in aluminium MMCs (159,160,193,194,213). Absence of dynamic recrystallization in the composites could be contributed to several factors. Firstly, hot workings of the composites are mostly conducted in a strain lower than 1 in each single step because of low ductility (220,224,225). The deformation is far too small to generate dynamic nucleation at large particles. It has been reported that dynamic recrystallization occurred in 5083 aluminium alloy which was deformed in strain 5 at the temperatures above 400°C in torsion (159). However, the same alloys containing 10% SiC particles did not show dynamic recrystallization during hot rolling in 25% reduction at 425°C (229).

Secondly, most aluminium MMCs studied in hot working contain low solutes, especially the alloys such as 6061 and 2014 Al. As mentioned in section 1-4-3-2, low solute could reduce possibility of dynamic recrystallization (151). Finally, if the transition theory could be applied, dynamic recrystallization is most unlikely to occur in the composites that deformed at high temperatures and low strain rates because no deformation zone or lattice rotation adjacent large particle can be produced in this condition (209,210).

Effect of large particle on recovered structure could be different in dependent on deformation temperature and strain rate (209). At low strain rates or high temperatures,
the microstructures are similar to those of the monolithic alloys. The size of subgrains both close to and away from the particles are similar to that of the monolithic alloys. At high strain rates or low temperatures, the microstructures are more irregular, and the subgrains near to the particles are smaller than those in the matrix. Generally, substructure in the composites is refined by the presence of high volume fraction of large particles (209,229).

Relations between subgrain size and deformation temperature, strain rate, and flow stress described by equations 1-4-10 and 1-4-11 for the composites have not been found to be reported.

1-5 The Purpose and the Scope of the Present Work

From review of the strengthening mechanisms of the short fiber and particulate metal matrix composites, it can be seen that strengthening of the short fiber and particulate composites is mainly due to microstructural changes in the matrix caused by the presence of the reinforcement instead of load transference between reinforcement and matrix. The most important microstructural change is that the matrix contains high density of dislocations introduced by difference in the thermal mismatch between reinforcement and matrix, and also by geometric necessary mismatch during deformation of the materials. This additional high density of dislocations not only enhance strength of the materials but also influence the microstructural development of the materials during thermomechanical processes. Any microstructural phenomena related to density and activation of the dislocation will be affected by the presence of reinforcement. These include precipitation, static restoration after deformation at room temperature, and dynamic restoration at elevated temperature. These processes, in turn, will affect mechanical properties of the composites. The purpose of the present
work is to study the behaviour of Al2O3 and SiC particles reinforced 6061Al alloys during ageing, cold working and annealing, and hot working in a 6061Al alloy. The work in these aspects is described as following:

I. Ageing Behaviour of Reinforced Materials

It has been known that ageing sequence of 6061 Al alloy is in order of formation of GP-I zone, β'' zone, β' and equilibrium Mg2Si phases. The presence of hard particles could accelerate ageing response of the alloy. However, from review of the publications, it can be noticed that it is still quite obscure in ageing characteristics of 6061Al alloy and its composites. Several questions are arisen:

1). Which stage of precipitation leads the alloy age hardened during artificial ageing, GP zones (GP-I or needle-shaped GP-II zones, β'') or rod-shaped β' phase.

2). How the reinforcements accelerate ageing response of 6061Al composites? Does the reinforcement affect formation and growth of GP zones or only accelerate formation of β' phase?

3). Effect of ageing temperature on the ageing sequence and the reasons for that the age hardening is accelerated in the composites at high temperature but not at low temperature need to be investigated.

4). Ageing sequence and precipitate structure in deformed alloys need to be studied.

The present work will employ isothermal and isochronal ageing assisted by DSC analysis and TEM microscopy to relate precipitation kinetics to precipitates morphology and hardness at different ageing temperature. Precipitate structure and its
orientation relationship with the matrix is not studied in this work since it has been reported (81-84). The purpose of this work is to relate morphology of the zones or precipitates to DSC analysis and hardness measurement, and to work out some answers for those questions mentioned above.

II. Cold Working and Static Recrystallization of the Composite

Recrystallization behaviour in the alloys containing particles is varied depending on particle size and volume fraction. Fine dispersions and precipitates may retard recrystallization by pinning dislocation and sub-boundaries. On the other hand, presence of large particles could accelerate recrystallization by stimulation of nucleation. In the materials containing both fine and large particles, recrystallization behaviour is depending on fraction of the two type of the particles but recrystallization kinetics are controlled by fine particles (129). However, there are still some aspects need to be studied, particularly in large ceramic particle reinforced materials. These aspects include;

1). Cold deformed structure of alloy containing high volume fraction of large particles should be studied in both scale of optical and electron microscopy. This could contribute understand of structural development in the materials during annealing. It can be expected that non-uniform straining in the matrix around large particles will occur and it will affect nucleation and recrystallization kinetics of the composites.

2). In cold working and annealing processes, reduction and annealing temperature are two main parameters that affect static restoration behaviour of alloys. It has been reported that strain level could affect nucleation rate on the particles in particle containing alloys but there is not much information about the effect of these parameters
on recrystallization of reinforced alloys, particularly the effect on recrystallized grain size.

3). Study of grain growth in the reinforced materials have not been reported, particularly abnormal grain growth during solution treatment. It is necessary to know the reaction between reinforcement and grain boundaries during high temperature treatment.

4) Solute and fine precipitates of alloying elements can retard nucleation and delay recrystallization in the deformed alloys, but limited information is available for 6061Al alloy. Effect of precipitates on restoration behaviour and grain structure of the reinforced 6061Al alloy needs to be investigated.

Therefore, cold working and annealing will be conducted with different reductions in both reinforced and unreinforced materials, and static restoration behaviour will be compared with the monolithic alloy. Recrystallized grain size and grain growth are also investigated. Effect of the precipitates will be studied by comparing the results of the material in annealed condition and solution treated condition.

III. Deformation Behaviour at Elevated Temperature

Hot working of the reinforced materials will be concentrated on following aspects:

1) Dynamic Restoration Behaviour of the Composite During Deformation at High Temperature
In static restoration, large particles act as nucleation sites and stimulate recrystallization of the composite. Whether the large particles could affect dynamic restoration in alloy containing high volume fraction of large particles is unknown. Restoration behaviour of the materials will be studied by plotting true stress-true strain curves and observing microstructures.

2) Flow Stress Dependent on Strain Rates and Temperatures

Strain rate sensitivity (m) or stress exponent (n') for both reinforced and unreinforced alloys will be evaluated according to relationship of flow stress and strain rate at various temperatures. Activation energy for deformation (Q) will be evaluated. These parameters will be used to correlate flow stress with temperature compensated strain rate (Z). High temperature strength of the materials will also be studied.

3) Microstructure Dependent on Strain Rate and Temperature

Deformed structure (grain or subgrain size) will be related to strain rate, temperature, and flow stress based on the experimental data, which will make a better understanding of microstructural development in reinforced material at high temperature.

4) Static Recrystallization Behaviour in Hot Worked Materials

Heat treatment following hot working is a common process in practice. The structure in heat treated materials will depend on hot working conditions such as deformation temperature, strain and strain rate. Even though some work has been reported in aluminium alloys and composites, effect of deformation temperature and strain rate on
the structure during following heat treatment has not been studied systematically (228). It is necessary to understand effect of dynamic restored structure on following static restoration in the reinforced materials and the role played by large reinforcement on structural development. Therefore, the study will be conducted by annealing of the samples deformed at different temperatures and strain rates and annealed structure will be related to deformation parameters.

The whole experiment is designed to reveal some thermomechanical characteristics of the reinforced materials. Mechanical testing followed by microstructure observation will be conducted in reinforced and unreinforced samples treated in different processes. The results and discussions will help us to understand these materials theoretically and practically, and it is also author's intention to provide some useful information for industries.
CHAPTER 2

MATERIALS AND EXPERIMENTS

2-1 Materials

The materials used in this work are three alloys; 1) 6061 aluminium alloy, 2) 6061 aluminium alloy containing 10 vol% of silicon carbide (SiC) particles (average size is 5μm in diameter), and 3) 6061 aluminium alloy containing 15 vol% of alumina (Al₂O₃) particles (average size is 25μm). SiC/6061Al alloy was supplied by Comalco Research Centre and Al₂O₃/6061Al alloy was obtained from Alcan (Canada) Ltd. Both materials are in the form of hot extruded bars. Chemical analysis of 6061 aluminium alloy is shown in Table 2.1.

Table 2.1 Composition of 6061 aluminium alloy (w%)

<table>
<thead>
<tr>
<th>Elements</th>
<th>Cu</th>
<th>Mg</th>
<th>Si</th>
<th>Fe</th>
<th>Cr</th>
<th>Mn</th>
<th>others</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>w%</td>
<td>0.25</td>
<td>0.82</td>
<td>0.6</td>
<td>0.15</td>
<td>0.1</td>
<td>0.12</td>
<td>&lt;0.05</td>
<td>balance</td>
</tr>
</tbody>
</table>

2-1-1 Microstructure of As-received Materials

Figures 2.1a and b show optical micrographs of Al₂O₃/6061Al and SiC/6061Al composites. It can be seen that, in general, the ceramic particles distribute in the matrix uniformly, despite slightly align along the direction of extrusion in Al₂O₃/6061Al composite. However, in SiC/6061Al composite, the particles are heavily aligned along
Fig. 2.1 Distribution of reinforcements in 6061Al matrix composites, a) SiC particle reinforced composite, and b) Al₂O₃ reinforced composite.

Fig. 2.2 The morphology of the reinforcements a) SiC particles and b) Al₂O₃ particles.
Fig. 2.3 TEM micrographs of reinforced materials, a) SiC/6061Al composite, and b) Al₂O₃/6061Al composite.
Fig. 2.4 Scheme of the sample for ageing cut from as-received material.

Fig. 2.5 Cylindrical sample for cold and hot compression tests.
the direction of extrusion, and some of the particulate free zones can be easily seen. Morphologies of the particles are shown in Figures 2.2a and b. Both Al₂O₃ and SiC particles show an irregular shape and rough surface which is suitable for gripping the matrix, and strengthening bond.

Figures 2.3a and b are TEM micrographs of the two as-received composites in which high dislocation density in the vicinity of SiC and Al₂O₃ particles can be noticed. It seems that a large number of dislocations was generated either by deformation during fabricating processes and/or as a result of strain generated due to mismatch in the thermal expansion coefficients between the ceramic particles and the matrix during cooling from fabricating temperature.

2-1-2 The Samples for Ageing and Compression Tests

Since the materials were supplied in a form of cylindrical bar, the samples for ageing were cut from the bar into the disc of 4 mm in thickness as shown in Fig.2.4. The samples used for cold and hot compression were machined to a cylinder in the size of 8 mm in diameter and 12 mm in length as shown in Fig.2.5. Axis of the cylinder is parallel to extrusion direction.

2-2 Experiments

Experimental studies were conducted in three stages; ageing, cold deformation and annealing, and high temperature deformation. The treatment of the specimens and experimental procedures are described in the following sections.
2-2-1 Isothermal and Isochronal Ageing

The specimens from Al₂O₃ and SiC reinforced and unreinforced materials were solution treated at 540°C for two hours then quenched in water. Solution treated specimens were kept in the furnace maintained at 125°C. Samples were aged successively for longer times from 1 to 680 hours. The materials were also aged at 170°C isothermally up to 50 hours. Isochronal ageing was conducted in a temperature range from 125 to 350°C for one hour. To ensure that these materials are treated at the same temperature, they were aged together. After ageing, the specimens were directly quenched in water. Ageing was conducted in air in a muffle furnace.

To investigate effect of pre-straining on ageing, Al₂O₃/6061Al alloy was deformed with 55% reduction after solution treatment, and then aged at 125°C up to 780 hours. The reason for choosing this low temperature is to avoid high level recovery which could significantly affect hardness measurements.

Hardness measurement was carried out using Vickers hardness test with 10 kg of load. The data were collected from at least 8 tests on each sample. Transmission electron microscopy was carried out to study microstructural changes in both reinforced and unreinforced specimens after ageing. At least three specimens in each alloy were observed, which were in the under-aged, peak-aged, and over-aged conditions. Ageing sequence was also monitored by differential scanning calorimetry (DSC).
2-2-2 Cold Deformation and Annealing

Two materials, Al₂O₃/6061Al and 6061Al were used in this experiment. Cylindrical samples were pre-treated in two different conditions prior to cold working. The samples were heat treated at 500 °C for one hour then cooled to room temperature in the furnace referring to condition OT. The samples were also treated at 540 °C for two hours, then water quenched referring to condition T4 (solution treated following natural ageing caused by delay during deformation at room temperature).

Pre-treated samples were compressed at room temperature in a servo-hydraulic testing machine in three reductions, 35, 55, and 70%. Nickel anti-seize tape was used as lubricant to avoid bulging which could assist to achieve uniform deformation. Cold worked samples were annealed between 200 and 500 °C in a muffle furnace for one hour. The samples were also annealed at 300 °C for up to 24 hours to determine the recrystallization kinetics. High temperature heat treatment was carried out at 540 °C for up to 40 hours on the samples given reduction of 55% for a grain growth study. All samples were water-quenched after heat treatment.

Recrystallization kinetics of the materials were monitored by hardness measurement. Deformed and annealed specimens were examined by means of standard techniques of optical microscopy using polarised light and transmission electron microscopy. Recrystallized grain size was determined on optical microscope using an eye piece with a cross line. Subgrain size was measured on TEM photos taken from at least two foils on each specimen.
Fig. 2.6 Computer aided servo-hydraulic testing machine with a furnace attachment; a) testing machine and control system, b) tools and water quench device.
Fig. 2.7 Schematic diagram of hot compression procedure.

Fig. 2.8 Samples before and after deformation.
2-2-3 Hot Working

Cylindrical samples machined from Al₂O₃/6061Al and 6061Al bars were pre-treated into the conditions of OT and T4 as described above. Uniaxial hot compression was conducted in a servo-hydraulic testing machine with a furnace attachment shown in Fig.2.6. The temperature range used in this test was from 200 to 500°C at an increment of 50°C. The specimens were compressed to a true strain of 0.9 at constant strain rates of 0.001 to 1 s⁻¹.

Schematic diagram of hot compression is shown in Fig.2.7. Prior to compression, the sample was heated up to deformation temperature and held for 5 minutes, then compressed to desired strain and quenched in water immediately after deformation. Nickel anti-seize tape was used as lubricant to minimise bulging of the sample during compression. The samples before and after being compressed are shown in Fig.2.8. It can be seen that the sample did not show any significant bulging indicating that the deformation was quite uniform. Testing temperature, strain, and constant strain rates were set up and controlled by computer. Load and extension were recorded by load cell and extensometer fixed on the machine. The data were collected by computer, and the true stress and true strain were plotted using these data.

Each deformed sample was cut into two pieces; one for structural examination and another piece was annealed for an hour at 500°C to study the static restoration behaviour in these hot deformed materials.
2-2-4 Structural Studies

Microstructures of the samples treated or tested as mentioned above were studied using optical microscopy and transmission electron microscopy (TEM). The optical microscopy was employed to observe general structure such as particle distribution, grain size and morphology. TEM was used to study the fine substructures such as dislocation network, precipitates, deformation microbands, and subgrain size.

2-2-4-1 Optical Microscopy

The specimens for optical microscopy were cut from the cold and hot deformed samples using diamond wheel. The surface which was observed is the one which is parallel to the compression axis. The specimens were mounted in epoxy resin for easy handling, and were prepared for metallographic observation by grinding through successively finer grades of 180, 240, 320, 400, 600, and 1200 SiC papers, using water as lubricant and coolant. The composite specimens were then subsequently polished on Textmet cloth impregnated with 6 micron diamond spray, and then 1 micron spray under adequate loading to allow the reinforcements and the matrix to be polished at similar rates and allow low relief polish. This was further followed by a polish on Microcloth carrying colloidal silica and water to remove scratches on matrix area in the composite. 6061 Al alloy was ground through similar grade papers. Diamond polishing was conducted on polishing cloth impregnated with 6 and 1 micron diamond sprays, respectively.

Electrolytic etching was carried out at a potential of 30 v DC in a Barker's etchant (5 g HBF₄ in 200 ml water) at room temperature. The specimens were anodised and viewed under polarized light in a Nikon optical microscope. It was found that etching
results were much better for monolithic 6061Al alloy than that for the composite. The grain size was measured using linear intercept method on the optical microscope directly. Eye piece with cross lines in a gauge was used to measure grains in two directions. The average grain size was estimated after counting at least 200 grains. It should be mention that there could be some error in the measurement of grain size on the composite specimens because the grain size was of about similar dimensions as that of Al₂O₃ particles, there was also some etching problem in some specimens.

2-2-4-2 Transmission and Scanning Electron Microscopy

The preparation of the specimens for TEM involved cutting thin slices from the sample from where the disc specimens of 3 mm in diameter were trepanned by spark cutting machine. The slices were cut in the direction parallel to the axis of cylindrical samples. The discs were first ground to 0.15 to 0.2 mm in thickness by grinding on 1200 grit SiC paper and were then electrolytically etched and polished using Struer Tenupol jet polishing system operating at 30 V in a solution of 20% HNO₃ in methanol at a temperature of -30 °C. The transmission electron microscopy was carried out using JEOL 2000 FX operating at 200 kV. Scanning electron microscopy was conducted in a HITACHI S-450 microscope operating at 20 kV to observe the morphology of the reinforcements.

2-2-5 Hardness Measurement

Conventional Vickers hardness test was chosen in this work instead of microhardness test on the matrix because it was very hard to avoid effect of reinforcement on indentation in the composites with high volume fraction of the particles, which could give a large variation in the hardness readings. The hardness measurements were
conducted on Vickers hardness testing machine using 10 kg load for aged samples and 5 kg load for annealed samples. Each hardness value taken was the representative of the average of at least eight readings. Hardness was measured immediately after the samples being deformed or heat treated to avoid any measurement affected by natural ageing.

2-2-6 Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry was used to study the effect of reinforcement on the ageing kinetics of the composites. The specimens were first solution treated at 540°C for two hours and quenched in water. The disc samples were analysed in a Mettler DSC30 from 25 to 500°C using a heating rate of 5°C/min. To examine microstructure in the alloys at exothermic peaks, the discs were also analysed from 25°C to identical peak temperatures and then cooled to 25°C at a cooling rate about 150°C/min. The discs were polished and etched for TEM microscopy.
CHAPTER 3

AGE HARDENING BEHAVIOUR OF SiC AND Al₂O₃ PARTICLES REINFORCED 6061 ALUMINIUM COMPOSITES

3-1 Introduction

6061 aluminium alloy is a type of age hardening alloy which could be age hardened naturally and/or artificially. Adding large volume fraction of particles or whiskers could affect ageing behaviour of the alloy. It has been found that existence of these reinforcements could accelerate ageing in the composite, and the hardness of reinforced alloys reached its peak value more quickly compared to the monolithic alloy during isothermal ageing at 170°C (52,58,59). However, accelerated ageing has not been observed in the reinforced materials when they are aged at temperature as low as 125° (52,58). It was considered that this is due to difference in ageing sequence at high and low ageing temperatures (52). Since limited information on microstructural investigation was available, the reasons for different ageing behaviour at different ageing temperatures were not clear. The purpose of the present work is to study the ageing behaviour of 6061Al alloy and its composites containing large SiC and Al₂O₃ particles at 125 and 170°C. The study was carried out by relating ageing structure to ageing curve and DSC analysis, that is, microstructural observations were correlated to changes in hardness and profile of DSC thermogram for identifying ageing sequence at the two temperatures. To understand precipitation kinetics of the alloys, the materials were also aged isochronally in the temperature range of 125 to 350°C for one hour.
Figure 3.1 Isothermal ageing curves for SiC/6061Al, Al₂O₃/6061Al, and 6061Al alloys at 125°C.

Figure 3.2 Isothermal age hardening curves for SiC/6061Al, Al₂O₃/6061Al, and 6061Al alloys at 170°C.
It has been suggested that straining prior to ageing could modify ageing sequence in 6061Al alloy because the formation of $\beta''$ zones could diminish whilst formation of $\beta'$ phase could be accelerated during ageing process (52,65,84). However, because of absence of microstructural investigation, no evidence could support the suggestion. In this work, $\text{Al}_2\text{O}_3/6061\text{Al}$ composite was aged at 125°C after given 55% reduction. The effect of straining on ageing sequence and ageing structure were studied using TEM and DSC analysis.

3-2 Experimental Results

3-2-1 Ageing Behaviour in Undeformed Materials

3-2-1-1 Age Hardening Curves

Hardness measurements of reinforced and unreinforced materials aged at 125 and 170°C are shown in Fig. 3.1 and 3.2, respectively. Several observations can be made. Firstly, it can be seen that hardness of the composites is much higher than that of the monolithic alloy after solution treatment. As the ageing time increases, hardness increases in both composites and monolithic alloy until the peak values are reached. Rate of increase in the hardness of reinforced and monolithic alloys are similar. Secondly, both reinforced and unreinforced alloys reach to their peak hardness after 480 hours of ageing at 125°C. No acceleration of ageing in the composites could be observed. On the other hand, these materials obtained their peak hardness much earlier when aged at 170°C. The two composites showed the maximum hardness in about 10 hours of ageing whilst the monolithic alloy took 28 hours to reach peak hardness. Thirdly, the hardness of SiC/6061 Al composite is higher than that of the $\text{Al}_2\text{O}_3/6061\text{Al}$ composite after solution treatment and also after ageing. The SiC
Figure 3.3 Isochronal age hardening curves for SiC/6061Al, Al₂O₃/6061Al, and 6061Al alloys from 25 to 350°C for one hour.
Figure 3.4 Thermograms of differential scanning calorimetric analysis for 6061Al, SiC/6061Al, and Al₂O₃/6061Al in a temperature range from 25 to 500°C at a heating rate of 5°C/min.
composite appears to show accelerated ageing leading to peak hardness at somewhat shorter time (10 hours) compared to $\text{Al}_2\text{O}_3$ composite (between 10 to 16 hours). Finally, the peak hardness is lower in the specimens aged at 170°C compared to that at 125°C, especially in the reinforced specimens; For example, peak hardness is 145 kg/mm$^2$ for SiC/6061Al composite aged at 125°C, but the peak value is about 140 kg/mm$^2$ while aged at 170°C.

The curves of hardness vs ageing temperature during isochronal ageing for the monolithic and the two reinforced materials are shown in Fig.3.3. The peak hardness was reached after one hour at 200°C in the two reinforced alloys and the monolithic alloy showed its peak hardness at 225°C. Accelerated ageing is obvious in the two composites, and rate of ageing in SiC composite is slightly higher than that in $\text{Al}_2\text{O}_3$ composite. The hardness decreases sharply in the two composites as the temperature increases above 225°C. The values of peak hardness for the three materials in isochronally aged condition are similar to that of isothermally aged alloys at 170°C.

3-2-1-2 Differential Scanning Calorimetric Analysis

To investigate precipitation kinetics in these materials, solution treated reinforced and unreinforced alloys were analysed using differential scanning calorimetric analysis (DSC). The DSC thermograms of SiC/6061Al, $\text{Al}_2\text{O}_3$/6061Al, and 6061 Al are shown in Fig. 3.4. There are two exothermic peaks (corresponding to precipitates formation) in each thermogram curve. The temperatures of initial reaction, first and second exothermic peaks for the three materials are listed in Table 3.1. It can be seen that the initial reaction temperatures for the three materials are quite similar. The two peak temperatures for the two composites are lower compared to that for the matrix alloy suggesting that the kinetics of precipitation are accelerated due to the presence of
Figure 3.5 Precipitate structures in 6061Al samples quenched from peak exothermic peak temperatures, a) quenched from first peak, and b) quenched from second peak.
the reinforcements in the composites. It also shows that the two exothermic peaks for SiC reinforced alloy are at lower temperatures compared to Al$_2$O$_3$ reinforced alloy indicating that precipitation in SiC/6061Al material is faster than in Al$_2$O$_3$/6061Al composite. SiC composite displays a higher heat capacity in first reaction compared to the other two materials.

Table 3.1 The initial reaction temperature ($T_i$), peak temperatures ($T_{p1,2}$) and heat capacities ($\Delta H_{1,2}$) of the three materials.

<table>
<thead>
<tr>
<th></th>
<th>6061Al</th>
<th>SiC/6061Al</th>
<th>Al$_2$O$_3$/6061Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_i$ (°C)</td>
<td>194</td>
<td>192</td>
<td>192</td>
</tr>
<tr>
<td>$T_{p1}$ (°C)</td>
<td>232</td>
<td>225</td>
<td>228</td>
</tr>
<tr>
<td>$\Delta H_1$ (J/g)</td>
<td>3.93 (180-260°C)*</td>
<td>5.61 (190-250°C)</td>
<td>4.50 (180-250°C)</td>
</tr>
<tr>
<td>$T_{p2}$ (°C)</td>
<td>280</td>
<td>268</td>
<td>274</td>
</tr>
<tr>
<td>$\Delta H_2$ (J/g)</td>
<td>1.28 (250-300°C)</td>
<td>1.53 (250-300°C)</td>
<td>1.28 (250-300°C)</td>
</tr>
</tbody>
</table>

$T_{p1}$ and $T_{p2}$: First and second exothermic peak temperatures. 
$\Delta H_1$ and $\Delta H_2$: Heat reaction capacities in the first and second peak regions. 
* Peak integration temperature range.

Precipitate structures in 6061Al alloy quenched from two exothermic peaks are shown in Fig.3.5a,b. The structure at first peak displays needle-shaped zones which were considered as $\beta''$ or GP-II zones, and the structure in second peak displays a coarse rod-shaped phase which was known as $\beta'$ phase (88). The structures in the present work are much coarser than that observed by Dutta (88) and the reason for this is that the heating rate in the present investigation is much lower (5 K/min) than that in the DSC studies of Dutta et al.
Figure 3.6 Quenched structure after two hours at 540°C, a) SiC/6061Al, b) Al₂O₃/6061Al, and c) 6061Al.
Figure 3.7 Microstructures of Al₂O₃/6061Al aged at 125°C for, a) 190 hours, b) 480 hours, and c) 680 hours.
Figure 3.8 Microstructures of Al₂O₃/6061Al aged at 170°C for, a) 1 hour, b) 6 hours, c) 10 hours, and d) 44 hours.
Figure 3.9 Microstructures of 6061Al alloy aged at 170°C for, a) 10 hours, b) 28 hours, and c) 50 hours.
3-2-1-3 Precipitate Structure

Figures 3.6(a,b) show dislocation structures in solution treated and water quenched reinforced materials. Dislocation density in the two composites is found to be higher than that in the monolithic alloy and SiC/6061Al exhibits a heavier dislocation network compared to Al₂O₃/6061Al composite. It can be seen that the dislocations are denser in the areas around large particles than in remote areas in the reinforced alloys.

Figures 3.7(a-c) show microstructures in the Al₂O₃/6061Al composite aged at 125 °C for 190, 480, and 680 hours respectively. Dot-shaped zones accompanied with a fine needle-shaped phase can be seen in the sample aged for 190 hours. When the sample achieves its peak hardness after 480 hours, most zones become fine needle-shaped. The density of the zones is much higher compared to the sample aged for 190 hours. The overaged sample shows a coarse needle-shaped phase after 680 hours of ageing at 125°C (Fig.3.7d). Development of the zones in the Al₂O₃ composite aged at 170 °C are shown in Fig.3.8(a-d). Images of the zones both dot and needle-shaped are very weak in the sample aged for an hour (Fig.3.7a). After 6 hours, the zones are mostly needle shaped. At the maximum hardness after 10 hours of ageing, Al₂O₃/6061Al composite displays dense and fine needle shaped zones (Fig.3.7c), whilst after 44 hours at 170°C, the alloy is overaged, and precipitate structure consists of needle-shaped zone and long rod-shaped phase as shown in Fig.3.8d. It can be noticed that the zones at this temperature are coarser than those in the sample aged at 125°C. The morphology of the zones and precipitates in 6061 Al monolithic alloy aged at 170°C are shown in Fig.3.9. It shows that development of the zones is similar to that in the composite aged at the same temperature but the times are much longer.
Figure 3.10 Precipitate structures of isochronally aged $\text{Al}_2\text{O}_3/6061\text{Al}$ material at the temperatures a) 170, b) 200, c) 225, and d) 300°C for one hour.
Figure 3.11 Age hardening curves of deformed and undeformed Al<sub>2</sub>O<sub>3</sub>/6061 Al alloy aged at 125°C.
Figure 3.12 Thermograms of differential scanning calorimetric analysis for deformed and undeformed Al$_2$O$_3$/6061Al alloy. Temperature range 25 to 500°C, heating rate 5°C/min.
Precipitate structure in isochronally aged $\text{Al}_2\text{O}_3$ composite is shown in Fig. 3.10. The needle-shaped structure in the sample with maximum hardness aged at 200°C is coarser than that in the sample aged at 170°C isothermally. As the temperature increases, the alloys overage quickly with the appearance of coarse rod-shaped phase at temperatures above 225°C.

3-2-2 Effect of Deformation on Ageing Behaviour of $\text{Al}_2\text{O}_3$/6061Al Composite

In order to study the effect of deformation on ageing behaviour, $\text{Al}_2\text{O}_3$/6061Al composite was given 55% reduction after solution treatment and then aged at 125°C isothermally up to 780 hours. DSC analysis and TEM microscopy were conducted to study ageing sequence and precipitation kinetics in the deformed and aged composite.

3-2-2-1 Ageing Curve and DSC Analysis of Deformed Composite

The effect of deformation on ageing behaviour can be seen from hardness curves as shown in Fig. 3.11. By comparison with undeformed material, hardness of deformed material prior to ageing is much higher caused by strain hardening. However, hardening rate is considerably low in deformed alloy during ageing at 125°C (increase in hardness is about 20 kg/mm$^2$ for deformed material but it is 40 kg/mm$^2$ for undeformed material after aged for 190 hours). Maximum hardness appears much earlier (190 hours) for the deformed sample compared to that for the undeformed composite (480 hours) but the hardness value is lower for the deformed alloy. The hardness of the deformed alloy decreases sharply after 190 hours.
Figure 3.13 Microstructure in deformed Al₂O₃/6061Al sample quenched from second exothermic peak temperature during DSC analysis.

Figure 3.14 Microstructure in deformed Al₂O₃/6061Al sample quenched from third exothermic peak temperature during DSC analysis.
Figure 3.15 Microstructures in isothermally aged deformed $\text{Al}_2\text{O}_3/6061\text{Al}$ alloy at $125^\circ\text{C}$ for, a) 190 hours and b) 780 hours.
DSC analysis of deformed composite is shown in Fig.3.12. Deformed sample displays three exothermic peaks. By comparison with undeformed alloy, the first peak is very small, and the first two peaks occur at 170 and 220°C. These temperatures are much lower than those for the undeformed alloy. The third peak is at about 365°C. Structure in the sample quenched from the second exothermic peak is shown in Fig.3.13. Coarse rod-shaped precipitates are present. Even the image of these precipitates is not as clear as that of undeformed samples, and distribution of the precipitates is non-uniform, they can still be identified as β' phase. The two exothermic peaks are thus caused by formations of β" and β' phases, respectively. Figure 3.14 shows microstructure of the sample quenched from the third exothermic peak. It shows that deformed structure has recrystallized partially suggesting that the third exothermic peak was caused by the recrystallization in the deformed composite.

3-2-2-2 Structure in Deformed Composite During Isothermal Ageing

The ageing structures in deformed samples are shown in Fig.3.15(a,b). Under developed cell structure with high density of dislocations is present but no β" zones and β' phase can be observed in peak hardened sample after 190 hours at 125°C (Fig.3.15a). However, long needle-shaped and rod shaped structures can be observed in over-aged sample after 780 hours even though they are not clearly displayed. It can be seen that a few needle shaped zones pointed by arrow in Fig.3.15b are similar to the structure in undeformed but overaged samples (Fig.3.7c and 3.9c), which are coarsened β" zones.
3-3 Discussion

3-3-1 Dislocation Structure in Solution Treated Composites

The results show that dislocation density in SiC and Al$_2$O$_3$ particles reinforced composites is higher than that in the monolithic 6061Al alloy after the materials have been solution treated and water quenched. These dislocations are preferentially distributed around large particles, (Fig.3.6a,b), suggesting that they are generated by the mismatch of the thermal expansion coefficients between the reinforcement and the matrix (40,47). The thermal stress caused by thermal mismatch is higher than the yield stress of the matrix alloy, and some of the stress will be released by generation of the dislocations. Both residual stresses and the dislocations may affect properties of the materials as well as subsequent ageing behaviour. It also can be noticed that dislocation density in SiC reinforced alloy is higher than that in Al$_2$O$_3$ composite. This is because of the fact that SiC has a smaller thermal expansion coefficient than Al$_2$O$_3$. The mismatch between the SiC particle and the matrix could be larger than that between the Al$_2$O$_3$ particle and the matrix. According to strengthening mechanism of the composites, this high density of dislocations can increase strength and hardness of the materials (25-33). Therefore, the two composites display much higher hardness than the matrix alloy after quenched from solution temperature and SiC particle reinforced alloy has higher hardness than Al$_2$O$_3$ composite.

3-3-2 Ageing Sequence in Particle Reinforced 6061 Al Composites

The shapes of the ageing curves for both reinforced and unreinforced materials are quite similar during isothermal and isochronal ageing at various temperatures. Even though the composites reach their peak hardness at a shorter time compared to the
monolithic alloy at 170°C, the composites exhibit a similar age hardening capacity as the monolithic alloy.

Structural observation of aged samples shows that there are three types of the zone structures in different morphologies, the dot, needle and rod shapes as shown in Fig.3.7b and 3.8b. These structures can be identified as GP-I, GP-II(β") and β' phases in sequential (62,83). It can be seen that the aged structures are dominated by the needle shaped zones in the three materials during ageing at both 125 and 170°C even though some dot shaped zone appears at 125°C (Fig.3.7a) and some coarse rod shaped phase presents in the overaged samples at 170°C (Fig.3.8d and 3.9d).

By correlating ageing structure in Fig.3.7-3.9 to the hardness measurement, it can be seen that hardness starts increasing when needle-shaped zones are formed. Before the peak hardness is reached, the zones are quite fine and density is low (Fig.3.7a and 3.8b). As ageing continues, the zones become denser and some of them become coarse while the hardness reaches peak value. Dot-shaped GP-I zones do appear in the sample aged at 125°C after a short time of ageing (Fig.3.7a) but they are found no responsibility for peak hardness because needle-shaped β" zones are already formed before peak hardness is reached. Continuing ageing causes coarsening of the β" zones, and leads to the materials overaged. Even though few coarse rod-shaped (β') precipitates are present in the alloys aged at 170°C for 44 hours (Fig.3.8d) and 50 hours (Fig.3.9c), age hardening and overage softening are found to be caused by formation and coarsening of the β" zones in the present ageing conditions. By comparison of aged structures in Fig.3.8 and 3.9, it can be seen that the ageing sequence in the reinforced material is similar to that in the monolithic alloy, and they both are age hardened by formation of the β" zones.
Table 3.2 Age hardening of three materials at different ageing temperatures

<table>
<thead>
<tr>
<th>Temperatures</th>
<th>Materials</th>
<th>$H_s$</th>
<th>$H_p$</th>
<th>$\Delta H$</th>
</tr>
</thead>
<tbody>
<tr>
<td>125°C</td>
<td>SiC/6061Al</td>
<td>104</td>
<td>146</td>
<td>42</td>
</tr>
<tr>
<td></td>
<td>Al$_2$O$_3$/6061Al</td>
<td>94</td>
<td>138</td>
<td>44</td>
</tr>
<tr>
<td></td>
<td>6061Al</td>
<td>75</td>
<td>117</td>
<td>42</td>
</tr>
<tr>
<td>170°C</td>
<td>SiC/6061Al</td>
<td>104</td>
<td>138</td>
<td>34</td>
</tr>
<tr>
<td></td>
<td>Al$_2$O$_3$/6061Al</td>
<td>94</td>
<td>129</td>
<td>35</td>
</tr>
<tr>
<td></td>
<td>6061Al</td>
<td>75</td>
<td>115</td>
<td>40</td>
</tr>
</tbody>
</table>

$H_s$ - Hardness of solution treated samples;
$H_p$ - Peak hardness of aged samples;
$\Delta H$ - Difference between $H_p$ and $H_s$.

Table 3.3 Age hardening in the three materials during isochronal ageing

<table>
<thead>
<tr>
<th>Materials</th>
<th>$H_s$</th>
<th>$H_p$</th>
<th>$\Delta H$</th>
<th>$T_p$</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC/6061Al</td>
<td>104</td>
<td>134</td>
<td>30</td>
<td>200°C</td>
</tr>
<tr>
<td>Al$_2$O$_3$/6061Al</td>
<td>94</td>
<td>123</td>
<td>29</td>
<td>200°C</td>
</tr>
<tr>
<td>6061Al</td>
<td>75</td>
<td>110</td>
<td>35</td>
<td>225°C</td>
</tr>
</tbody>
</table>

$H_s$, $H_p$, and $\Delta H$ have the same meanings as above.
$T_p$ - The temperature at which the material is aged to peak hardness.
3-3-3 Effect of Ageing Temperature on Ageing Behaviour of 6061Al Alloy and Its Composites

The peak hardness in both reinforced and unreinforced materials were reached after 480 hours at 125°C, but it only took 10 hours for the composites and 28 hours for the monolithic alloy to get their peak hardness at 170°C. It is obvious that increase in age temperature accelerated ageing of the alloys. This can be understood as that diffusion rate increases as the temperature increases, and which in turn enhances both nucleation and growth rates of the β" zones.

As discussed in previous section, age hardening in the present conditions is dominated by formation and growth of the β" zones. Acceleration of the ageing at high temperature could be caused by high nucleation and growth rates of the zones, but increase in growth rate seems to be more pronounced as the temperature increases. This can be observed from zone structures in two peak hardened samples aged at 125 and 170°C. The zone structure in the sample aged at 125°C is much finer and denser than that aged at 170°C suggesting that growth of the zones is faster at 170°C. The occurrence of the fast growth of the zones reduces the ageing time required to reach the peak hardness significantly but this occurs at the cost of hardening capacity. This can be seen from the data in Tables 3.2 and 3.3. The values of ΔH (difference between solution treated hardness and peak hardness) for both composites and 6061Al alloy are smaller in samples aged at 170°C than that in samples aged at 125°C indicating higher growth rate of the zone at higher temperatures. Before density of the zones reaches highest level, most zones are coarsened. Coarse zones give the alloys low hardness. This is why the peak hardness appears faster but is lower as age temperature is high in most researches (52,58,75).
It has been reported that increase in ageing temperature could change ageing sequence in Al alloys (52,58,75). The DSC analysis in the present study shows that the zones are different at different temperatures, that is, the $\beta''$ zones form at about 220°C (the morphology is shown in Fig.3.5a) whilst $\beta'$ zones appear at 270°C (Fig.3.5b). The results from isochronal ageing also show that the structures are different in the samples aged at 200°C from that aged at 300°C (Fig.3.10). However, within the certain temperature range, ageing sequence is not changed. For instance, in a temperature range between 125 and 170°C, the ageing structures are quite similar (mainly $\beta''$ zones) even the zones in the sample aged at 170°C are coarser compared to that in the samples aged at 125°C. Thus, in the temperature range of 125-170°C, ageing sequence could be considered as unchanged.

3-3-4 Effect of Reinforcement on Ageing Behaviour of the Composites

The results show that presence of large volume fraction of ceramic reinforcements does not change ageing sequence in the composites in the present ageing conditions. However, the composites aged isothermally at 170°C as well isochronally between 125-350°C do show accelerated ageing. DSC analysis shows that two exothermic peaks for formation of $\beta''$ and $\beta'$ in the composites move to lower temperature compared to the monolithic alloy suggesting that both formations of $\beta''$ and $\beta'$ phases are accelerated in the composites. However, accelerated ageing is not obvious when the composites are aged at 125°C. Both reinforced and unreinforced alloys reach their peak hardness after 480 hours at 125°C. It seems that effect of reinforcements on the formation and growth of the zones is diminished at the low temperature. This could probably be explained as follows.
Increase in diffusivity can be resulted from increasing temperature but also from straining. Even though the internal strains generated from thermal mismatch could be released partially by generation of dislocations, there are still some strains left. These residual strains and dislocations could increase diffusivity and thus enhance nucleation rate and growth rate of the zones and intermetallic precipitates. However, it seems that addition of high density of dislocations and residual strains have mainly increased growth rate of the zones during ageing at 170°C. This is because that nucleation of the zones can be accelerated but once no more nuclei can be formed, high diffusivity through the dislocations and internal strains will be only benefit for growth of the zones. This can be observed from DSC thermograms which show that the composites exhibit quite similar initial reaction temperatures but lower peak temperatures compared to the monolithic alloy. It can be said that as the reaction (precipitation) starts, it finishes at a shorter time in the composites, which could be due to high growth rate of the zones. High growth rate of the zones in the reinforced material can also be related to difference in $\Delta H$ values given in Table 3.2. It can be seen that $\Delta H$ is lower for the composites (34 and 35) compared to the matrix alloy (40) when the both are aged at 443K. Lower $\Delta H$ in the reinforced material seems due to inability to obtain maximum hardness during ageing. Such a behaviour, in fact, is due to a higher coarsening rate of the zones, which in turn decreases its ability to acquire maximum hardness during ageing at 443 K.

However, at low temperature (125°C) both nucleation and growth rates are low because low diffusivity of solute elements. In addition to this, the dislocation activity is limited at low temperature, which in turn leads to a limited effect on ageing. The formation and growth of the zones in the reinforced materials may therefore be not very different from that in the monolithic alloy at low temperature. It can be seen that both the composite and the monolithic alloys attain their peak hardness at same time.
and ΔH values are similar (Table 3.2). This indicates that growth rate of the zones is quite similar in the two materials when they are aged at such low temperature. As mentioned in the previous section, ageing sequences in 6061Al alloy and its composite at both 125 and 170°C are similar and the hardening agent is 𝛽" zone at these temperatures. The accelerated ageing of the reinforced alloy observed at 170°C is due to increased rates of formation and growth of 𝛽" zones at higher temperature and not as a result of change in the ageing sequence.

It should be mentioned that the zone structures observed at peak hardness for both reinforced and monolithic alloys are similar and they both are identified as 𝛽" zones. The accelerated ageing in the composites is thus not because of formation of 𝛽' phase but instead due to increase in nucleation and growth rates of the 𝛽" zones. It should be also mentioned that the zones seems to distribute unevenly in the matrix, and growth of the zones is also quite non-uniform (Fig. 3.5a, 7a-c and 8a-c). This indicates that growth of the 𝛽" zones are preferable faster in some regions than the others. Faster growth of the zones in these regions were probably caused by dislocations even though it was considered that the zones nucleated uniformly throughout the matrix.

From ageing curves in Fig.3.1 and 3.2, it can be seen that ageing rate of SiC composite is slightly higher compared to Al₂O₃ composite at the two temperatures, and the time required to reach peak hardness is shorter for SiC composite compared to Al₂O₃ composite at 170°C. DSC thermograms also show the acceleration of both 𝛽" and 𝛽' phases in SiC reinforced material compared to Al₂O₃ composite. Evidently, the formation of the zones is enhanced by higher dislocation density and residual strain introduced by SiC particles. Furthermore, SiC composite has a smaller interparticle spacing which probably also contributes to higher ageing rate compared to Al₂O₃ reinforced material.
3-3-5 Ageing Behaviour of Deformed Al2O3 Composite

The overall effect of deformation on ageing behaviour of the alloy generally involves contribution from two processes - age hardening as well as recovery of deformed structure. Firstly, formation of the zones and the precipitates during ageing can lead to age hardening. On the other hand, recovery occurring in deformed material will result in decrease in the hardness. Thus, the ageing curve of deformed material could be considered as a result of combination of these two processes leading to a low age hardening rate. However, microstructure of deformed sample in the present work did not exhibit significant recovery even after 780 hours at 125°C. Cell structure with high density of dislocations are still remained. If precipitation hardening is not concerned, this substructure will give the alloy a higher hardness compared to the undeformed alloy which contains non-strained grain structure. However, in the present work, undeformed alloy with well developed grain structure has higher hardness than deformed alloy after ageing 190 hours. This indicates that a lower peak hardness for the deformed material compared to undeformed material is due to degradation of age hardening capability instead of recovery.

DSC thermogram of deformed composite shows that first exothermic peak occurred at 170°C is so small that it can be hardly seen. The second peak which appears at 220°C overlaps with the first peak, and it emerges at a quite lower temperature even compared to the first peak in undeformed material. As discussed earlier that the first peak relates to β" zone whilst the second peak corresponds to β' phase, which means that the formation of both β" zone and β' phase is accelerated but accelerated formation of β' phase is more significant. It seems that formation of β" zone is depressed by formation of β' phase. Since β" zone is the only hardening agent in this alloy, reduction or absence of β" considerably or totally diminish hardening ability in
the material. This could be the reason that deformed sample displays less hardening compared to undeformed material.

Suppression of β"
 zones in deformed material has been considered as that the zone forming elements, Si and Mg, are trapped by the tangled dislocations after heavy cold deformation (65). It has also been considered as a result of acceleration of nucleation and growth of intermediate phase when deformed alloy is aged at certain high temperatures (95). However, from DSC analysis in the present study, it was found that formations of both β" and β' phases were accelerated but the β' phase was formed so rapidly that it started forming well before the complete formation of β" zones occurred. Generally, β" zones are uniformly nucleated in the matrix prior to β' phase. When diffusivity increases by means of high temperature or heavy strain, formation of β' phase can be considerably accelerated and it will reduce formation of β" zones since dislocations are considered as more favourite nucleation sites for β' phase (95). Furthermore, the growth of the β" zones is also accelerated by dislocations which act as diffusion channels. High coarsening rate of the zones will also contribute to low peak hardness in deformed material.

Evidently, the effect of deformation on ageing behaviour is different from that of reinforcements. The β" zones are not suppressed by the presence of reinforcement in reinforced material but by deformation. This is because dislocation density or other defects are much higher in deformed material compared to in undeformed reinforced material, which act as diffusion channels. In addition of these defects, deformation bands could also act as diffusion channels leading to considerably high coarsening rate of β" zone and high formation rate of β' phase in deformed alloy.
It should be noted that $\beta''$ zones cannot be observed in the peak hardened sample, and $\beta'$ phase can only be seen in overaged samples after 780 hours with obscure image (Fig. 3.15). It seems that the contrast of $\beta''$ and $\beta'$ phases under TEM were disturbed or diminished by heavily dislocated structure. As ageing time increased, a certain degree of annihilation and rearrangement of dislocations might occur and image of precipitates became visible.

3-4 Summary

1. The reinforced materials have similar age hardening ability as the monolithic alloy. Age hardening in the alloy is achieved by formation of GP zone, and maximum hardness is contributed by fine needle-shaped $\beta''$ phase (or called GP-II zone) during artificial ageing at temperatures of 125 and 170°C. Overageing with hardness decrease is caused by coarsening of the $\beta''$ phase.

2. The presence of large particle doesn't change ageing sequence but can accelerate formation and coarsening of the $\beta''$ phase, and subsequently, ageing is accelerated. This is resulted from high dislocation density and residual stress introduced by reinforcement in the composites. However, this can only be observed while the alloy is aged at certain high temperature (170°C). When the temperature is low (125°C), formation and coarsening of the $\beta''$ phase is considerably slow, and accelerated ageing in reinforced alloys is hardly observed.

3. DSC thermograms show that introduction of the large particles can decrease formation temperature of $\beta''$ zones and $\beta'$ phase. Even initial reaction temperature for $\beta''$ zones is similar in both reinforced and unreinforced materials, the temperature of exothermic peak is lower for the reinforced materials compared to the unreinforced
alloy. This indicates that accelerated ageing is mainly caused by fast coarsening of the \( \beta'' \) zones in the composites.

4. Age hardening capability could be degraded by straining prior to ageing. This is because deformation prior to ageing reduces the formation of \( \beta'' \) zones which are the only hardening agent in this alloy. Coarsening of \( \beta'' \) zones and formation of \( \beta' \) phase are accelerated because of high density of dislocations and deformation bands introduced by straining leading to accelerated overageing and lower peak hardness in reinforced materials compared to undeformed alloy.
CHAPTER 4

RESTORATION BEHAVIOUR AND GRAIN GROWTH OF COLD-WORKED Al2O3/6061Al COMPOSITE

4.1 Introduction

The effect of particles on static restoration behaviour on the aluminium alloys has been studied by many researchers. Most of the studies were conducted on the alloys containing small volume fraction of dispersoid fine particles (97-114). Even though some investigations have been carried out to study the recrystallization behaviour of the materials containing both large particles and fine dispersoids, little information has been published on how the presence of a high volume fraction of large particles influences the restoration behaviour of reinforced materials (26, 27, 40-45). The present study was, therefore, carried out to investigate the effect of large particles on the deformation structure, recrystallization behaviour and recrystallized grain size in a 6061Al matrix composite reinforced with 15% volume fraction of Al2O3 particle of ~25 μm in size. Both reinforced and monolithic alloys with OT* condition were cold deformed with reductions of 35, 55, and 70%, and then annealed in a temperature range from 200 to 500°C for one hour. The materials were also deformed with reduction of 55%, and annealed at 300°C for up to 24 hours. Particle stimulated recrystallization and recrystallization kinetics were studied. Effect of reduction and annealing temperature on recrystallized grain size was also investigated.

* OT - Materials were annealed at 500°C for one hour, then slowly cooled in the furnace.
Figure 4.1 Optical microstructures of cold deformed Al$_2$O$_3$/6061Al composite with reductions of, a) 35, b) 55, and c) 70%.
Figure 4.2 TEM micrographs of the deformed Al₂O₃/6061Al composite with reductions of, a) 35, b) 55, and c) 70%.
To study effect of solute and precipitate on recrystallization behaviour and grain size in the reinforced alloy, the material was also pre-treated in T4** condition prior to cold deformation and annealing. Recrystallization kinetics and recrystallized grain size were compared to that of the material in OT condition. The grain growth in OT treated reinforced and monolithic alloys was studied by annealing the samples at 540°C for up to 40 hours after given 55% reduction.

4.2 Experimental Results

4.2.1 Deformation Structure

Figures 4.1(a - c) show optical micrographs of cold-deformed Al2O3/6061Al composite (OT condition) with reduction of 35, 55, and 70%, respectively. Grains are elongated in the sample given 35% reduction, but individual grain is still identifiable (Fig.4.1a). However, after the sample being deformed with 70% reduction, the grains are heavily elongated and deformation bands or shearing bands can be clearly seen (Fig.4.1b).

TEM micrographs of the deformed Al2O3/6061 Al composite are shown in Fig.4.2(a - c). It shows that a deformation band, in fact, consists of a large number of very fine microbands. These bands are about 0.1 to 2 μm in width depending on the amount of deformation. The banded structure is very pronounced in a highly deformed sample and they become obscure in the sample with low reduction (Fig.4.2a). Mostly the ** T4 - Alloys were solution treated at 540°C for 2 hours followed by water quench, then leaved at room temperature for hours before cold working (delayed during cold working)
Hardness (Hv 5kg)

Annealing Temperature (°C)

Al2O3/6061Al, 35% reduction

Annealing Temperature (°C)

Al2O3/6061Al, 55% reduction
Figure 4.3 Hardness as a function of annealing temperature for Al₂O₃/6061Al composite with three reductions of a) 35, b) 55, and c) 70%, (annealed for an hour).
6061Al, 35\% reduction

Annealing Temperature (°C)

Hardness (Hv 5kg)

6061Al, 55\% reduction

Annealing Temperature (°C)

Hardness (Hv 5kg)
Figure 4.4 Hardness as a function of annealing temperature for 6061Al alloy with three reductions of a) 35, b) 55, and c) 70%, (annealed for an hour).
bands are needle-shaped and vary in length. Within the band, there is cell structure which contains a high density of dislocations. The cell structure is elongated in the ratio of about 2 to 1 along the length of the bands. The width of the microbands in both reinforced and unreinforced alloys given different reductions is listed in Table 4.1. It shows that the presence of large Al₂O₃ particles reduces the width of the microband. The width of the bands decreases as reduction increases from 35 to 55%. As reduction further increases, the size of the bands varies little in both reinforced and unreinforced alloys.

Table 4.1 Microband width in Al₂O₃/6061Al and 6061Al alloys given different reductions

<table>
<thead>
<tr>
<th>Reductions</th>
<th>Al₂O₃/6061Al (µm)</th>
<th>6061Al (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>35%</td>
<td>1 - 2.5</td>
<td>1 - 3</td>
</tr>
<tr>
<td>55%</td>
<td>0.2 - 0.6</td>
<td>0.3 - 0.7</td>
</tr>
<tr>
<td>70%</td>
<td>0.1 - 0.5</td>
<td>0.2 - 0.5</td>
</tr>
</tbody>
</table>

4-2-2 Effect of Annealing on Hardness

Results are plotted in Fig. 4.3(a - c) and 4.4(a - c) as hardness versus annealing temperature for Al₂O₃/6061Al and 6061 Al alloy with three reductions, respectively. Hardness of the composite with 35% reduction decreases as annealing temperature increases until it reaches its lowest value at around 400°C. Whilst the lowest hardness for the composite with 55 and 70% of cold deformation is found at 350°C. The
35% reduction

\[ \text{Annealing Temperature } ^\circ C \]

55% reduction

\[ \text{Annealing temperature } ^\circ C \]
Figure 4.5 Normalized hardness as a function of annealing temperature for both \( \text{Al}_2\text{O}_3/6061\text{Al} \) composite and 6061Al monolithic alloy with three reductions of, a) 35, b) 55, and c) 70%.

Figure 4.6 Isothermal annealing curves for both \( \text{Al}_2\text{O}_3/6061\text{Al} \) composite and 6061Al alloy at 300°C after given 55% reduction.
Figure 4.7 Hardness as a function of annealing temperature for T4 condition pre-treated alloys, a) Al$_2$O$_3$/6061Al composite, and b) 6061Al alloy.
hardness of the 6061 Al alloy increases slightly when annealed at 200°C but then decreases sharply as the annealing temperature increases (Fig.4.4). Both the reinforced and unreinforced alloys exhibit increase in hardness when annealed above 450°C suggesting that the dissolution of the precipitates has occurred above this temperature.

Normalized hardness at different annealing temperatures is calculated using $H_i / H_0$, where $H_i$ is the instantaneous hardness measured after annealing at particular temperature and $H_0$ is the hardness of as deformed material. The results are shown in Fig. 4.5(a - c) for comparison between $\text{Al}_2\text{O}_3/\text{6061Al}$ composite and 6061Al alloy at three different reductions. It can be seen that curves for the composites move to a lower temperature, and the temperature of fully recrystallization is ~50°C lower than that for the monolithic alloy with identical reduction.

Recrystallization kinetics of the reinforced and unreinforced 6061Al alloys have been studied by isothermal annealing. Fig.4.6 shows the curves of hardness versus annealing time for the $\text{Al}_2\text{O}_3/\text{6061Al}$ and the monolithic alloys given 55% of cold reduction followed by annealing at 300°C. The reinforced alloy reached its lowest hardness after about 4 hours at 300°C, but 6061Al alloy had not obtained its lowest value even after 8 hours of annealing indicating that recrystallization kinetics have been accelerated in the reinforced material.

The materials pre-treated in T4 condition have been annealed isochronally after given 55% of reduction. The hardness curves for the composite and 6061Al alloy are shown in Fig.4.7(a,b). By comparing to the results in Fig.4.3 and 4.4, it can be seen that the composite in both OT and T4 conditions reaches to lowest hardness values at about
Figure 4.8 Optical Microstructures of $\text{Al}_2\text{O}_3$/6061 Al composite given 35% reduction and annealed at a) 200, b) 300, c) 400, and d) 500°C for an hour.
Figure 4.9 Optical Microstructures of Al₂O₃/6061Al composite given 55% of reduction and annealed at a) 200, b) 250, c) 350, and d) 400°C for an hour.
Figure 4.10 Optical Microstructures of 6061Al alloy given 55% of reduction and annealed at a) 300, b) 350, c) 400, and d) 500°C for an hour.
350°C, whilst the temperature is 400°C at which the monolithic alloy exhibits its lowest hardness.

4-2-3 Microstructural Development during Annealing

Optical micrographs in Fig.4.8a-d show the microstructure of Al$_2$O$_3$ composite given 35% reduction and annealed between 200 - 500°C. There is no visible difference in the microstructure of samples annealed up to 250°C. Difference is, however, observed in the sample annealed at 300°C (Fig.4.8b), in which some fine equiaxed recrystallized grains can be found. As the temperature increased further, the volume fraction of equiaxed grains increased. Recrystallization was almost complete in the sample given 35% of reduction at 400°C. The samples given 55 and 70% of reduction were found to start recrystallization at 250°C and exhibit completed recrystallization structure while annealed at 400°C. Figures 9a-d show annealed structures in the composite given 55% of cold deformation. The structure in the sample annealed at 200°C is similar to deformed structure indicating that no significant restoration occurs. At 250°C, some equiaxed grains are present, and as annealing temperature increased to 400°C, deformed structure is fully recrystallized to equiaxed grain structure (Fig.9b,d). The structures in monolithic alloy given 55% of reduction followed by annealing in a temperature range of 300 to 500°C are shown in Fig.4.10. Few small equiaxed grains can be observed in sample annealed at 300°C. As temperature increases, more recrystallized grains are present. The sample annealed at 500°C exhibits a fully recrystallized structure (Fig.4.10d).

Figures 4.11(a,b) show TEM micrographs of Al$_2$O$_3$/6061Al specimens given 55% reduction followed by annealing at 200 and 250°C. By comparison with deformed structure, annealed sample at 200°C shows that the cells increase in size and cell walls
Figure 4.11 TEM micrographs of Al$_2$O$_3$/6061Al composite annealed at, a) 200, and b) 250°C, after given 55% of reduction.

Figure 4.12 TEM micrographs of 6061Al alloy annealed at, a) 200, and b) 250°C after given 55% of reduction.
Figure 4.13 Optical micrographs of T4 Al$_2$O$_3$/6061Al given 55% of reduction followed by annealing at, a) 200, b) 250, c) 350, and d) 500°C.
Figure 4.14 TEM micrographs of T4 - Al₂O₃/6061Al composite annealed at, a) 200, and b) 300°C after given 55% of reduction.
become thinner. As annealing temperature increases, the cell structure is developed to subgrains with sharp grain boundaries at 250°C. Subgrains can be seen clearly in some area adjacent to Al₂O₃ particles in the composite. Figure 4.12(a,b) is a group of TEM photographs that show substructural development in the monolithic alloy given same reduction as the composite. It can be observed that substructures in 6061 Al alloy is not well developed compared to that in the composite.

The structural development in the composite in T4 condition during annealing following 55% of cold deformation is shown in Fig.4.13(a-d). Banded structure is present in the sample annealed at 200°C suggesting that recovery is not significant and recrystallization does not occur. Few equiaxed grains adjacent to large particles can be observed in the sample annealed at 250°C as shown in Fig.4.13b indicating that recrystallization has started. As the annealing temperature increases to 350°C, banding structure disappears (Fig.4.13c) and massive equiaxed grains are exhibited suggesting that deformed structure is almost fully recrystallized. The micrographs in Fig.4.14a and b show the recovered substructures in T4 Al₂O₃/6061Al annealed at 200 and 300°C. Plate-shaped precipitates can be observed in recovered substructure, but no interaction between precipitates and cell walls or sub-boundaries can be found.

**4-2-4 Nucleation of Recrystallization in the Composite**

Figures 4.15(a,b) show micrographs that display nucleation of recrystallization at large particles. First recrystallized grain was found in the composite annealed for one hour at 250°C (Fig.4.15a). It was found that all nuclei were associated with large particles at this temperature, and they developed preferentially in the narrow gaps between the large particles. As the annealing temperature increases, more recrystallized grains can be seen in the composite. The sample of composite annealed
Figure 4.15 Recrystallization nucleation in Al$_2$O$_3$/6061Al composite given 55% of reduction, a) annealed at 250°C, and b) annealed at 300°C for 1 hour.

Figure 4.16 Recrystallized grains in T4- Al$_2$O$_3$/6061Al composite given 55% of reduction, a) annealed at 250°C, and b) at 300°C for 1 hour.
Figure 4.17 Recrystallized grains in 6061Al alloy given 55% of reduction and annealed at 300°C for 1 hour. (foil 1 and 2)
Figure 4.18 Recrystallized structure in a) Al₂O₃/6061Al composite and b) 6061Al alloy. The materials were annealed at 500°C for 1 hour after 70% of cold compression.
at 300°C displays the structure which is with mostly recrystallized new grains, and only the region far from large particles remains non-recrystallized fine subgrains (Fig.4.15b). In the present study, most nuclei are found to be at the large particles even though nucleation can also occur on pre-existing grain boundaries and deformation bands. Figures 4.16a and b show recrystallized grains in T4 treated composite samples annealed for one hour at 250 and 300°C after given 55% of reduction. It is found that all new grains are associated with large particles. Plate-shaped precipitates can be seen in both recrystallized grain and non-recrystallized area, but no grain boundary pinning can be observed (Fig.416b).

Figures 4.17a and b are TEM micrographs taken from annealed 6061 Al alloy given 55% reduction. Few nuclei can be observed in the sample annealed at 300°C for one hour, and these nuclei are found to be connected with large inclusion particles or particle groups present in the matrix.

4-2-5 Recrystallized Grain in Reinforced Material

The recrystallization finishing temperature (RFT) is determined from the hardness vs temperature curves shown in Fig.4.4 and 4.5, and observation of the optical microstructures. It is found that RFTs for reinforced and unreinforced alloys are 400 and 450°C respectively in the samples deformed at 55 and 70% reductions. Figures 4.18a, and b show typical recrystallized structures in Al₂O₃/6061Al composite and 6061 Al alloy that were annealed at 500°C for one hour after given 70% cold compression. It is found that grains in the composite are quite equiaxed with a size similar to the Al₂O₃ particles, but grains in the monolithic alloy are elongated parallel to the deformation bands. Recrystallized grain size for both reinforced and unreinforced alloys is listed in Table 4.2. The results show that the composite has
Figure 4.19 Effect of annealing temperature on recrystallized grain size in both reinforced and unreinforced 6061Al alloys given 55% of reduction.

Figure 4.20 Effect of reduction on recrystallized grain size in reinforced and unreinforce 6061Al alloys after the alloys were annealed at 500°C for 1 hour.
Figure 4.21 Grain size as a function of annealing time in Al₂O₃/6061Al composite and 6061Al alloy at 540°C.
Table 4.2 Recrystallized grain size in Al$_2$O$_3$/6061Al and 6061Al alloys given different reductions

<table>
<thead>
<tr>
<th>Materials</th>
<th>Mean initial grain size (μm)</th>
<th>Reduction (%)</th>
<th>Annealing temperature (°C)</th>
<th>Mean recrystallized grain size (μm)</th>
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<tr>
<td>6061Al</td>
<td>105</td>
<td>35</td>
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<td>400</td>
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<tr>
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<td></td>
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<td>500</td>
<td>17.31</td>
</tr>
</tbody>
</table>

Table 4.3 Comparison of recrystallized grain sizes of Al$_2$O$_3$/6061Al in OT and T4 conditions

<table>
<thead>
<tr>
<th>Annealing temperature (°C)</th>
<th>Reduction (%)</th>
<th>Grain size (μm)</th>
</tr>
</thead>
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<tr>
<td></td>
<td></td>
<td>OT condition</td>
</tr>
<tr>
<td>400°C</td>
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<td>18.84</td>
</tr>
<tr>
<td>450°C</td>
<td>55%</td>
<td>18.64</td>
</tr>
<tr>
<td>500°C</td>
<td></td>
<td>18.15</td>
</tr>
</tbody>
</table>
finer average recrystallized grain size than matrix alloy under identical testing conditions.

The plots of grain size vs annealing temperature for reinforced and unreinforced materials deformed with 55% reduction are shown in Fig.4.19. It shows that the recrystallized grain size decreases slightly with increasing temperature in both the composite and the matrix alloy. Furthermore, the effect of temperature on grain size is more pronounced in the high temperature range. For example, when temperature is increased from 500 to 540°C, the grain size decreases from about 18 to 15 μm in the composite and from 31 to 27 μm in the monolithic alloy.

The effect of reduction on recrystallized grain size is shown in Fig.4.20. The grain size after annealing at 500°C decreases from 34 to 25 μm in the monolithic alloy and from 20 to 17 μm in the Al₂O₃/6061Al composite when reduction increases from 35 to 70%. These results suggest that the degree of deformation does not have significant effect on grain size in the reinforced material compared to the matrix alloy.

Comparison of the recrystallized grain size in OT and T4 conditions is listed in Table 4.3. It shows that the material with T4 condition has finer recrystallized grain size than the OT tempered material under identical deformation and annealing conditions.

4-2-6 Grain Growth in Reinforced Material

Grain growth at high temperature is studied by annealing the samples at 540°C up to 40 hours after given 55% of reduction. Grain size against annealing time for cold deformed Al₂O₃/6061Al and 6061Al alloys is plotted in Fig.4.21. It can be seen that average grain size in the composite increases from 15.5 to 21μm when annealing time
Figure 4.22 Optical micrographs of Al$_2$O$_3$/6061Al composite given 55% of reduction and annealed at 540°C, a) for 1 hour, and b) for 40 hours.
Figure 4.23 Optical micrographs of 6061Al alloy given 55% of reduction and annealed at 540°C, a) for 1 hour, and b) for 40 hours.
increases from 1 hour to 40 hours. On the other hand, grain size of the monolithic 6061 Al alloy increases from 26.6 to 43.2 μm under the same treatment condition, exhibiting faster growth rate. Optical micrographs of the composite and 6061Al alloy annealed at 540°C for 1 hour and 40 hours are shown in Fig.4.22 and 4.23, respectively. The reinforced material shows little grain growth after 40 hours at 540°C. However, abnormal grain growth can be observed in the monolithic alloy after 40 hours at 540°C (Fig.4.23).

4.3 Discussion

4-3-1 Deformed Structure in the Composite

Deformation of the composite is quite different to the single phase alloy, since the composite contains a high volume fraction of large non-deformable ceramic particles which can significantly influence deformation behaviour of the material. One of the characteristics is non-uniform deformation of the matrix around the large particles. This can be seen in Fig.4.2 which shows a particle interrupting the flow of deformed matrix. It seems that large particles act as obstacles to plastic deformation of the matrix. If it is supposed that no debonding occurred at the interface of Al₂O₃/Al, then the gradients of deformation will be built up in the matrix around particles and an increasingly dense array of dislocations has to be stored around hard particles to accommodate the gradients of deformation (100). Also, the large particles can increase the compatibility constraints imposed upon the matrix and this will further interfere with the banded structure, resulting in an increase of localized lattice rotation, especially in the vicinity of the Al₂O₃ particles.
Another phenomenon is that deformed substructure in the composite is finer than that in the matrix alloy suggesting that matrix of the composite experienced higher constrained deformation than the unreinforced alloy since the hard ceramic particles did not deform plastically in the composite. Such a situation can lead to a high density of dislocations and large lattice rotations in the matrix around the Al₂O₃ particles which will eventually influence the restoration behaviour of the composite (120).

4-3-2 Nucleation of Recrystallization in the Reinforced Alloy during Annealing

Nucleation of recrystallization in deformed metals is preferentially associated with the sites where large misorientations have developed during deformation such as grain boundaries, deformation bands and deformation zones around large particles. It has been shown that the presence of large particles could considerably increase nucleation sites for recrystallization (109-111, 113-117). In the present study, all recrystallized grains observed in the composite, at early stages, are associated with the large particles. Recrystallized new grains are first observed in the composite annealed at 250°C for 1 hour (Fig.4-15). These new grains are found preferentially at the large particles, especially in the narrow gaps between the large particles. It is known that the matrix around large particles is likely to have large misorientation and dense dislocation network resulting from the geometrically necessary dislocation array (50). According to Humphreys’ studies (113,119,120), the inhomogeneous plastic deformation at large particles (>1μm) will be accommodated by local lattice rotations which are dependent on particle size and strain (Fig.1.7). In the present conditions (the average particle size is 25μm and strains are 0.35, 0.55 and 0.7), the misorientation caused by lattice rotation could be more than 30° in the areas around the
Figure 4.24 Deformation model of single crystal bonded with large particles, a) non-deformed and b) after deformation by shearing (Ashby) (50).

Figure 4.25 Another form of deformation model of single crystal bonded with large particles, a) non-deformed, b) deformed by shearing.
particles. Those areas will preferentially become nuclei of recrystallization (113,120,147).

However, it was found that not all particles had a same ability to stimulate nucleation to the same extent. Some particles were found to associate nuclei in lower temperature than the others. This can be explained using Ashby's single crystal composite model (50). Ashby model is shown in Fig.4.24. Particles are spaced apart in a ductile single crystal matrix, in which the lattice plan is vertical to particle surface (Fig.4.24a). Suppose that matrix is strongly bonded, that is, no sliding occurs in the plane of interface, and the composite shears by slip in the matrix on single slip system in a direction normal to the particle surface. Lattice rotation occurs, and geometrically necessary dislocations build up in the area adjacent to particles (Fig.4.24b). However, if considering an alternate case in which the lattice plane is parallel to the particle surface as shown in Fig.4.25a, lattice deformation will be different from that in former case. The lattice rotation related to the particle surface is small and density of dislocation will be low (Fig.4.25b). Even though deformation is operated by multi-slip system in Al alloy and deformation of a polycrystalline sample is much more complicated, these two models can give an idea that some large particles generate lattice rotation in the matrix nearby while some particles can not, depending on orientation relationship of matrix grains and particle surface.

Closely spaced large particles or particles group showed stronger stimulating ability of nucleation. This phenomenon may also be explained by Ashby model in which lattice rotation increases as particles space decreases for the same amount of shear, that is, lattice rotation of the matrix material within the particle group is generally larger than the area surrounding a single particle.
By comparison, nucleation of the recrystallization in the monolithic alloy can not be observed at 250°C. First recrystallized grain was found in sample annealed at 300°C which is 50°C higher in recrystallization start temperature RST compared to reinforced alloy. These nuclei in the matrix alloy were also found to be connected to large inclusions or inclusion clusters. However, most nuclei seem to be on the deformation bands or previous grain boundaries because of limited inclusions.

4-3-3 Effect of Reinforcement on Recrystallization Kinetics of the Alloy

The results show that recrystallization finish temperature for reinforced alloy is about 50°C lower than that for the monolithic alloy at three different reductions (Fig.4.5). Optical micrographs show that reinforced material possessed equiaxed grain structure after annealed at 350°C for 1 hour, but the monolithic alloy displayed a partially recrystallized structure until the sample was annealed at 400°C for 1 hour. Acceleration of recrystallization was also found in isothermal annealed samples. Deformed reinforced material reaches to its lowest hardness in 4 hours at 300°C, but it took 14 hours for the monolithic alloy to achieve its lowest value.

Acceleration of recrystallization kinetics in the reinforced material is mainly resulted from particle stimulating nucleation. The large particles have priority as nucleation sites, and this will decrease start temperature of recrystallization. The grains nucleated at large particles grow by consuming deformation zones around the particles (Fig.4.15). The recrystallization completed as new grains grew towards remote area of the matrix until the grain boundaries contacted each other or contacted to other large particles. The path of migration of the grain boundary is shortened because of high density of nuclei and restriction of the particle spacing. Therefore, acceleration of
recrystallization kinetics is caused by two reasons. Firstly, particle stimulating recrystallization reduces nucleation temperature and incubation period, and secondly the time or temperature necessary for completing recrystallization are decreased because the path of migration of grain boundary is shorter.

4-3-4 Recrystallized Grains in the Composite

The presence of the large Al₂O₃ particles not only affects recrystallization kinetics but also affects recrystallized grain size. The grain size in fully recrystallized composite could also be different depending on reduction, annealing temperature, and heat treatment prior to deformation.

4-3-4-1 Effect of the Large Particles on Recrystallized Structure

Introduction of the large Al₂O₃ particles has a grain refinement effect due to the large particles act as nucleation stimulus. From Table 4.2, it can be seen that the composite possesses smaller grain size than the monolithic alloy at identical reduction and annealing temperature. However, the refinement could be limited by interparticle spacing of the large particles. It is considered that if the particles act as nucleation sites, the recrystallized grains will have a size comparable with the interparticle spacing (120). The composite used in the present work contains 15% vol. of Al₂O₃ particles of 25 µm in average diameter. The interparticle spacing is calculated by using Humphreys' technique and is found to be about 27µm (110). The grain size listed in Table 4.2 ranges from 15 to 21 µm depending on cold deformation and annealing temperature. A possible explanation for the grain size being smaller than the particle spacing is that the grains could grow from both sides of the particles resulting in two or three grains sharing the deformation zone between the particles as shown in
Figure 4.26 Several grains share interparticle spacing in the reinforced alloy annealed at 500 °C for 1 hour after given 55% of reduction.
Fig. 4.26 (annealed at 500°C three particles share spacing). Whatever the mechanism by which grains grow, grain size will be restricted by the interparticle spacing.

4-3-4-2 Effect of Annealing Temperature on the Grain Size

Recrystallized grain size is influenced by annealing temperature in two ways. On one hand, increasing annealing temperature can increase nucleation rate. It has been reported that the rate of low-angle boundary migration rate can increase relative to the high-angle boundary migration rate with increasing temperature resulting in higher nucleation rate relative to the growth rate (147,148). On the other hand, with temperature increasing, fine precipitates will grow coarser and lose some of the capability of grain boundary pinning resulting in high growth rate. If the former effect is strong, recrystallized grains would become finer at higher temperature. In the present work, effect of the annealing temperature on the grain size can be seen from Fig.4.19 (plot of grain size vs temperature) which shows that recrystallized grain size decreases as annealing temperature increases in both reinforced and unreinforced alloys. Grain refinement is more obvious when temperature is increased from 500 to 540°C. The average grain size in the samples deformed with 55% reduction is reduced from 18.15 to 15.42 μm for the composite and from 31 to 26.6 μm for the matrix alloy. These observations suggest that nucleation rate seems to be increasing at high temperature in these alloys.

4-3-4-3 Effect of Reduction on the Grain Size

Generally, as reduction increases, nucleation rate will be increased and recrystallized grain size will be refined. The present results show that grain size in both reinforced and unreinforced alloys decreases as reduction increases. Deformed structure shows
that original grains are heavily distorted and deformation bands and shearing increase as reduction increases. This heavily distorted structure will increase nucleation sites and refine recrystallized grains. However, effect of reduction on recrystallized grain size seems to be not significant in the composite compared to the monolithic alloy (Table 4.2). The reason for such behaviour seems due to the fact that nucleation is mostly occurred on the deformation bands in the monolithic alloy, and density of the bands increases as reduction increases. The nucleation rate will be enhanced, and thus the recrystallized structure is considerably refined in the monolithic alloy. By contrary, nucleation in the composite is determined by density of the reinforcement since mostly nucleation occurs at large reinforcement particles. Even though reduction could have some effect on the nucleation rate in the composite, it is not significant as in the monolithic alloy.

It can be noticed that the grain size of the monolithic alloy is refined from initial size of 105\(\mu m\) to about 33\(\mu m\) after given 35\% reduction but the grain size of the composite only changes from 21 to about 18\(\mu m\) even after give 70\% reduction. It is well known that final grain size is dependent on initial grain size and reduction in the monolithic alloys (134-p122). However, it seems that the theories for evaluation of recrystallized grain size in the monolithic alloy can not be applied to the composite. This is probably because of the special way of nucleation in the composite, which is mainly occurred at large particles. Even though the fine initial grain size and high constrained deformation can significantly increase dislocation density and deformation bands in the matrix of the composite, nucleation rate may not be increased largely because these nucleation sites such as deformation bands and initial boundaries have low priority for nucleation compared to the large particles. As mentioned above, in the reinforced material, nucleation rate is controlled by large particles and final grain size is restricted by
Figure 4.27 Restoration index against annealing temperature for the reinforced alloy in OT and T4 conditions; restoration index \( I_r = (H_0 - H_i) / (H_0 - H_r) \), where \( H_0 \), \( H_i \), and \( H_r \) are the hardness of as deformed, instant, and fully recrystallized samples, respectively.
interparticle spacing. This is the reason that grain size of the composite is not significantly affected by initial grain size, reduction and annealing temperature.

4-3-5 Effect of Pre-treatment Conditions on Recrystallization Behaviour and Grain Size of the Reinforced Alloy

The effect of pre-treatment conditions on recrystallization was studied by comparison of recrystallization behaviour in the samples with OT and T4 conditions. In the material with O tempered condition, the alloying elements, Si and Mg, were present mostly in the form of coarse precipitates. Whilst, alloy elements in T4 treated material were in the state of solid solution. These soluted alloying elements could affect deformed structure which in turn could influence recrystallization by solute drag and precipitate pinning during annealing. However, the results show that both materials start softening and reach to their lowest hardness at similar temperature. No much difference in recrystallization kinetics between T4 and O tempered composite has been found, except high initial hardness and rapid drop in hardness due to precipitation in T4 tempered material. If using restoration index to display restoration procedure, it can be found that the material in the two conditions exhibits similar behaviour (Fig.4.27). Optical micrographs show that the recrystallization of the composite in the two conditions starts at similar temperature (250°C), and complete temperature is also similar (400 °C). This indicates that solution treatment and subsequent precipitation have no effect on the recrystallization kinetics.

The precipitates in 6061Al alloy and it composite are mainly plate-shaped Mg$_2$Si and some spherical Si. As mentioned in Chapter 3, Mg$_2$Si phase is developed from GP-I, GP-II zones or $\beta''$ and $\beta'$ phases. Within these phases, only $\beta''$ play an important role in age hardening. However, the formation of $\beta''$ is at a quite low temperature, at 200°C
Figure 4.28 Recrystallized structure in the reinforced alloy with T4 condition, annealed at 350°C for 1 hour after given 55% of reduction.
for 1 hour in undeformed sample as shown in Fig.3.10b. DSC analysis also showed that precipitation temperature was much lower than the recrystallization temperature, suggesting that, before recrystallization started, precipitates have coarsened (Fig.3.12,14) and lost pinning ability. In deformed sample annealed at 300°C for 1 hour, coarse plate-shaped Mg2Si precipitates have been found, and these incoherent plate-shaped precipitates did not interact with dislocations and cell walls (Fig.4.14b). As temperature increased, recrystallization occurred. Like O tempered material, recrystallized grains were associated with large particles and grew by consuming the matrix around the particles. Precipitates have been found in both recrystallized grains and unrecrystallized structure as shown in Fig.4-16b. No grain boundary pinning could be observed suggesting that those precipitates have no effect on recrystallization leading to similar recrystallization kinetics in the material with OT and T4 conditions.

However, the sample in T4 condition exhibited finer recrystallized grains (Fig.4.13d) compared to the sample in OT condition (Fig.4.9d). This seems to be caused by high nucleation rate in T4 material. High nucleation rate is probably caused by solute pinning effect in T4 treated material. The solute drag could increase density of dislocation and lattice distortion significantly during deformation leading to solution hardening (15,150,160). The high density of dislocations and large lattice rotation may enhance nucleation at large particles as well as increase possibility of nucleation at previous grain boundaries. Figure 4.28 (T4, 350°C, 55%) shows recrystallized grains in T4 treated composite annealed at 350°C for 1 hour. It can be seen that there are several grains in the matrix between the large particles indicating that nucleation of recrystallization also occurred at the sites such as deformation bands or previous grain boundaries. Therefore, evidently nucleation rate is higher in T4 treated samples compared to O tempered samples. Solute pinning would be released by precipitation during annealing at the temperature above 250°C. Increase in the movement and
rearrangement of the dislocation could further enhance nucleation rate in T4 treated materials.

It has been known that fine recrystallized grains could be due to a result of precipitate pinning during annealing. Dislocation and grain boundary pinning could retard nucleation and recrystallization kinetics. The incubation of nucleation enables more grains to nucleate, and in subsequent, fine recrystallized grains can be obtained (131). This refinement of grains is in the cost of incubation of recrystallization. This is not a case of the present work since recrystallization is not postponed in T4 materials and no pinning could be found. Therefore, it is evident that solution treatment prior to cold deformation has no much effect on recrystallization kinetics but could refine grains of the alloy by increasing nucleation rate during subsequent annealing.

4-3-6 Grain Growth in the Composite

To investigate grain growth after recrystallization, deformed reinforced and unreinforced materials were heat treated at 540°C up to 40 hours. In fact, this temperature is considered to be a solution-treating temperature for 6061 aluminium and its composites (230).

The results show that both reinforced and unreinforced materials contain equiaxed fine grains after one hour at 540°C. After 40 hours, abnormal grain growth has been found in monolithic alloy (Fig.4.23b). Some grains were encroaching upon others and became larger in size. This abnormal grain growth is hard to be found in the reinforced alloy (Fig.4.22b). It can be seen that even few grains are larger than others, they are restricted in an area surrounded by the large particles as arrow pointed in Fig.4.22b. Grain growth only occurred in the interparticle spacing. As the grain
Figure 4.29 Grain boundaries are pinned by particles in the specimens annealed at 540°C for 40 hours, a,b) composite and c) matrix alloy.
boundaries contacted to large particles, further growth was stopped. This restriction of
reinforcement in the composite allows grain growth at a very low level compared to
the monolithic alloy. This can be seen in Fig. 4.21 which shows grain growth rate is
higher in the monolithic alloy than that in the composite.

Nevertheless, the grain growth in both materials is not significant after 40 hours at this
high temperature. It seems that grain growth after primary recrystallization was
inhibited. There are two possible reasons for inhibition of grain growth in present
materials and treatment conditions: solute-induced and particle-induced inhibitions
(134). Alloying elements could be mostly in solution after 40 hours at 540°C and
solute drag may reduce or stop migration of grain boundaries. The particles, including
reinforcement and inclusions may also pin grain boundaries and inhibit movement of
the boundaries. TEM micrographs (Fig. 4.29a-c) show grain boundaries pinned by
particles of ~ 1μm diameter in both reinforced and unreinforced alloys treated at 540°C
for 40 hours. Those particles are mainly Fe, Mn, and Si bearing inclusions and fine
Al₂O₃ particles in the matrix of the composite. It is envisaged that these large inclusion
particles play an important role in retardation of grain growth by restricting boundary
movement in the composite. There is similar amount of inclusions in the monolithic
alloy, and they also pin grain boundaries. However, since absence of fine and large
Al₂O₃ particles, restriction of grain growth in the monolithic alloy is not efficient as
that in the reinforced alloy.

4-4 Summary

1. The presence of the large Al₂O₃ particle could result in non-uniform deformation in
the matrix area around the large particles resulting in a high density of microbands and
dense dislocation networks during cold deformation in the composite.
2. During recrystallization, the large Al₂O₃ particles act as nucleation sites for recrystallization, especially closely-located particle groups which have a strong capability to simulate recrystallization.

3. High nucleation rate caused by (large) particle stimulation can accelerate the recrystallization kinetics in the composite resulting in a lower recrystallization temperature compared to the matrix alloy.

4. Recrystallized grain size is mainly controlled by the interparticle spacing of the large particles in reinforced material even though the amount of reduction and annealing temperature have some effect on grain structure. Finer grains are present in the reinforced material resulted from high nucleation rate. Also, the composite shows a more equiaxed and uniformly distributed grain structure after recrystallization in comparison with the matrix alloy.

5. The material in T4 condition displays a much higher hardness compared to OT alloy after cold deformation. This is caused by solute drag during deformation, which considerably increases density of dislocations and nucleation rate during following annealing in T4 alloy. The high nucleation rate leads to a finer grain size in T4 alloy compared to OT alloy during recrystallization.

6. Grain growth in the composite at high temperature (540°C) is not significant because of restriction of interparticle spacing. Migration of the boundaries can also be pinned by inclusion particles leading to an insufficient grain growth in both reinforced and unreinforced materials.
CHAPTER 5

DEFORMATION BEHAVIOUR AND MICROSTRUCTURAL DEVELOPMENT OF $\text{Al}_2\text{O}_3$ PARTICLE REINFORCED 6061Al ALLOY AT ELEVATED TEMPERATURE

5.1 Introduction

One of prime advantages of the aluminium MMCs is that the billets of the composites can be deformed to needed shape and size by conventional secondary fabrication processes such as extrusion and rolling. Like other metallic materials, these deformation processes are generally conducted at elevated temperatures and thus it is important to understand deformation behaviour and microstructural development of the particulate reinforced aluminium alloys during deformation at high temperature. Extensive research work has been carried out on the hot working behaviour of particle containing alloys but most studies were conducted on dispersion strengthened alloys that containing small volume fraction and fine particles (159,162,206,208,214-217). High temperature research on the particle or short fibre reinforced MMCs have been carried out to study the creep behaviour which is generally conducted at extremely low strain rates (226-228). Limited investigations have been undertaken on deformation behaviour and structural development of the composites at conventional strain rates employed in industry. The present work was thus carried out to study the deformation behaviour, microstructural development and their relationship in $\text{Al}_2\text{O}_3$ particle reinforced 6061Al alloy. In this chapter, the strength of the reinforced material at high temperature, effect of temperature and strain rate on the flow behaviour and microstructure based on experimental results are discussed.
True Stress (MPa)
True Stress (MPa)
Figure 5.1 True stress - true strain curves at different temperatures for Al₂O₃/6061Al composite at constant strain rates of a) 1 s⁻¹, b) 0.1 s⁻¹, c) 0.01 s⁻¹, and d) 0.001 s⁻¹.
True Strain

Strain Rate 0.1/s

True Stress (MPa)
True Stress (MPa)

True Strain

Strain rate 1/s

Legend:
- ▲ 450°C
- ▼ 400°C
- ○ 350°C
- ■ 300°C
- □ 250°C
- ○ 500°C
Figure 5.2 True stress - true strain curves at different temperatures for 6061 Al alloy at constant strain rates of a) 1 s$^{-1}$, b) 0.1 s$^{-1}$, c) 0.01 s$^{-1}$, and d) 0.001 s$^{-1}$.
The hot deformation experiments were conducted on hot compression machine aided with computer. Cylindrical samples were compressed in a temperature range of 250 to 500°C at constant strain rates of 0.001 to 1 s\(^{-1}\). After deformation, the samples were water quenched immediately. To study the recrystallization microstructure in hot worked composite, deformed samples were annealed at 500°C for one hour. The effect of deformation temperature and strain rate on recrystallized structure and grain size was discussed.

5 - 2 Experimental Results

Hot compression experiments were conducted on \(\text{Al}_2\text{O}_3/6061\text{Al}\) composite and 6061Al alloy in OT condition. Limited hot deformation testing was also carried out on these materials in T4 condition to study the effect of dynamic precipitation on the deformation behaviour.

5-2-1 True Stress - True Strain Curves

True stress true strain curves at constant strain rates of 1 x 10\(^{-3}\) to 1 s\(^{-1}\) in a temperature range between 250 and 500°C (one test was conducted at 200°C and at a strain rate of 0.1 s\(^{-1}\)) for \(\text{Al}_2\text{O}_3/6061\text{Al}\) alloy are shown in Fig.5.1(a - d). It can be seen that the shape of the curve is characterized by parabolic hardening at 200°C, but at the temperatures above 250°C, the curves are flattened after an initial stage of strain hardening. These curves are typical of the form expected when dynamic recovery occurs; that is, the flow stress rises sharply to a maximum and then approaches a steady-state value. Stress-strain curves for monolithic 6061Al alloy tested at various temperatures and strain rates are shown in Fig.5.2(a-d). The shape of the curves is similar to that of the reinforced material. It should be noted that real curves obtained...
Figure 5.3 Typical true stress - true strain curve for Al₂O₃/6061Al and 6061Al alloys at elevated temperature
Figure 5.4 Plots of $\ln \sigma$ against $\ln \dot{\varepsilon}$ for, a) $\text{Al}_2\text{O}_3/6061\text{Al}$ composite, and b) 6061Al alloy.
during testing (Fig.5.3) contain noise signals that probably came from testing machine and control system. However, the noises have no effect on identification of flow behaviour of the alloys.

5-2-2 Effect of Strain Rate and Temperature on the Flow Stress

ln\(\sigma\) vs ln\(\dot{\varepsilon}\) plots for Al\(_2\)O\(_3\)/6061Al composite and 6061Al monolithic alloy are shown in Fig.5.4(a-b) in which linear curve was obtained for both reinforced and unreinforced alloys. The linear feature of the ln\(\sigma\)-ln\(\dot{\varepsilon}\) curves allows the \(\sigma-\dot{\varepsilon}\) relationship to be represented by the following empirical equation:

\[
\sigma = K\dot{\varepsilon}^m \tag{5-1}
\]

where \(m\) is the apparent strain rate sensitivity exponent which is the slope of the curve in Fig.5.4. The \(m\) values for both reinforced and unreinforced alloys are listed in Table 5.1.

Combined effect of strain rate and deformation temperature on flow stress under hot-working conditions can be described by following equation:

\[
\dot{\varepsilon} = A_1[\sinh(\alpha \sigma)]^{n'}\exp\left(\frac{Q}{RT}\right) \tag{5-2}
\]

where \(A_1\), \(\alpha\), and \(R\) are constants that are independent of temperature and stress. \(\dot{\varepsilon}\) is constant strain rate, \(\sigma\) is flow stress (maximum true stress at flow curve) and \(T\) is deformation temperature. \(n'\) is stress exponent and \(Q\) is an experimental activation energy for plastic deformation, also taken as stress and temperature-independent (154,155). To plot the data according to Eq.5-2, the values of \(\alpha\), \(n'\) and \(Q\) must be
Figure 5.5 Plots of $\ln \dot{\varepsilon}$ versus $\ln [\sinh (\alpha \sigma)]$ for, a) $\text{Al}_2\text{O}_3/6061\text{Al}$ composite, and b) $6061\text{Al}$ alloy. (Slope of the curve is $n'$).
Figure 5.6 Plots of $\ln[\sinh(\alpha \sigma)]$ against $1/T$ for, a) Al$_2$O$_3$/6061Al composite, and b) 6061Al alloy, activation energy for deformation, $Q$, were evaluated from slope of the curves.
known. Calculations of $\alpha$ and $n'$ are described in Appendix 1. The values of $\alpha$ were 0.03 for Al$_2$O$_3$/6061Al composite, and 0.035 for 6061Al alloy. The corresponding values of stress exponent $n'$ were 5.1 and 5 for the composite and the monolithic alloy, respectively.

<table>
<thead>
<tr>
<th>Temperature</th>
<th>6061Al (OT)</th>
<th>Al$_2$O$_3$/6061Al (OT)</th>
</tr>
</thead>
<tbody>
<tr>
<td>250°C</td>
<td>0.05</td>
<td>0.0504</td>
</tr>
<tr>
<td>300°C</td>
<td>0.0825</td>
<td>0.7480</td>
</tr>
<tr>
<td>350°C</td>
<td>0.1134</td>
<td>0.1008</td>
</tr>
<tr>
<td>400°C</td>
<td>0.1319</td>
<td>0.1257</td>
</tr>
<tr>
<td>450°C</td>
<td>0.152</td>
<td>0.1416</td>
</tr>
<tr>
<td>500°C</td>
<td>0.1675</td>
<td>0.1566</td>
</tr>
</tbody>
</table>

The experimental data for hot compression of the composite and 6061Al alloy are plotted in Fig. 5.5(a - b) using the values of $\alpha$ determined above. The data are plotted in a form of ln$\dot{\varepsilon}$ vs ln[$\sinh(\alpha \sigma)$]. It shows that the data are fitted on parallel straight lines with the slope of $n'$ suggesting that relationship of stress, strain rate, and temperature in hot compression for both reinforced and unreinforced alloys could be described by Eq.5.2. Figures 5.6a and b show relationship between ln[$\sinh(\alpha \sigma)$] and 1/T, and the activation energy (Q) for deformation could be evaluated from the slope of these curves. It is found that average value of the Q is 222 kJ/mole for Al$_2$O$_3$/6061Al composite, and 204 kJ/mole for 6061Al alloy. A temperature-modified strain rate or Zener-Hollomon parameter can be written as following:

$$Z = \dot{\varepsilon} \exp\left(\frac{Q}{RT}\right) = A_1[\sinh(\alpha \sigma)]^{n'}$$ (5 - 3)
Figure 5.7 Flow stresses are related to temperature compensated strain rate through a plot of $\ln Z$ vs $\ln[\sinh(\alpha \sigma)]$ for Al$_2$O$_3$/6061Al composite.

Figure 5.8 Flow stresses are related to temperature compensated strain rate through a plot of $\ln Z$ vs $\ln[\sinh(\alpha \sigma)]$ for 6061Al alloy.
Figure 5.9 True stress - true strain curves for Al$_2$O$_3$/6061Al pre-treated in T4 condition composite, a) at a constant strain rate of 0.1 s$^{-1}$, and b) at temperature 400°C and constant strain rates from 0.001 to 1 s$^{-1}$. 
Figure 5.10 True stress - true strain curves of 6061Al alloy pre-treated in T4 condition at a constant strain rate of 0.1 s\(^{-1}\).
Figure 5.11 Proof stress as a function of deformation temperature at different strain rates for a) Al$_2$O$_3$/6061Al, and b) 6061Al alloy.
Figure 5.12 Plots of proof stresses against strain rates in a temperature range from 250 to 500°C for, a) Al₂O₃/6061Al composite, and b) 6061Al alloy.
Flow stress and deformation parameters (strain rate and temperature) for the composite are plotted as \( \ln Z \) vs \( \ln[\sinh(\alpha \sigma)] \) as shown in Fig.5.7 whilst a plot for 6061Al alloy is shown in Fig.5.8. The figures show that the flow stress increases as strain rate increases and deformation temperature decreases.

5-2-3 Effect of Pre-treatment Conditions on the Flow Stress

The samples of \( \text{Al}_2\text{O}_3/6061\text{Al} \) in T4 condition were tested in a temperature range of 200 to 500°C only at a strain rate of 0.1 s\(^{-1}\). However, at 400°C deformation was carried out in the strain rate of 0.001 to 1 s\(^{-1}\). The resulting true stress-true strain curves are shown in Fig.5.9(a - b). The figures show that the flow stress decreases rapidly after it reaches a maximum value at the temperatures of 250 and 300°C. As deformation temperature increases (350-450°C), the flow curves tend to be flatter, that is, the decrease of the flow stress is marginal after it reaches a maximum value. At 500°C, the curve is flat. This behaviour is also found in 6061Al alloy in T4 condition as shown in Fig.5.10.

5-2-4 Effect of Temperature and Strain Rate on the Proof Stress

Since both reinforced and unreinforced alloys exhibited no obvious yield point in the flow curves, the stress at the point corresponding to 0.2% plastic strain is defined as proof stress or yield stress. Figures 5.11 a and b show proof stress as a function of deformation temperature at different strain rates for reinforced and unreinforced alloys. The proof stresses in both materials decrease as deformation temperature increases smoothly. Proof stress vs strain rate in a temperature range 250 to 500 °C is plotted in Fig.5.12(a-b) for these materials. At low temperature (250°C), proof stress is less dependent on strain rate whilst at high temperature (500°C), it shows much large
Figure 5.13 The schematic diagram of determination of initial strain hardening rate from true stress-true strain curve.
Figure 5.14 Initial strain hardening rate as a function of deformation temperature for, a) reinforced alloy, and b) unreinforced alloy.
Figure 5.15 Effect of strain rate on strain hardening rate at different temperature, a) the composite, and b) the monolithic alloy.
increase in the stress with the strain rate. Both reinforced and unreinforced alloys exhibited linear relationship between $\ln \sigma_{0.2}$ and $\ln \dot{\varepsilon}$.

5-2-5 Effect of Temperature and Strain Rate on Strain Hardening

Strain hardening rate or work hardening rate can be expressed as $\frac{\partial \sigma}{\partial \varepsilon} |_{\varepsilon,T}$ while the relationship between stress and strain is defined as $\sigma = f(\varepsilon)$. Where $\sigma$ is true stress and $\varepsilon$ is true strain (plastic strain). From the true stress - true strain curve (Fig.5.3), it can be seen that above proof stress point, the material exhibits rapid strain hardening, after which hardening rate decreases as straining continues. Strain hardening rate at low strain is defined as initial strain hardening rate, and it could be evaluated by $\Delta \sigma / \Delta \varepsilon$ at low strains (0.01-0.02) in a small increment of $\Delta \varepsilon$. In the present work, $\Delta \varepsilon$ is evaluated to be 0.005 by intercept technique (Fig. 5.13).

The initial strain hardening rates calculated by above technique for both reinforced and monolithic materials have been plotted as a function of temperature at various strain rates as shown in Fig.5.14 (a - b). It can be seen that strain hardening rates decrease as temperature increases. At strain rate of 1 s$^{-1}$, the hardening rate decreases more rapidly compared to that at strain rate of 0.001 s$^{-1}$, which indicates that the temperature seems to have more effect on strain hardening at higher strain rate than at lower strain rate.

Plots of strain hardening as a function of strain rate for both the composite and the matrix alloy are shown in Fig.5.15 (a-b). It can be seen that strain hardening rate increases as strain rate increases for both reinforced and unreinforced materials. However, hardening rates are more sensitive to strain rate in low temperature range (250 and 300 °C) as observed.
Figure 5.16 Microstructures in Al₂O₃/6061Al composite deformed in a temperature range from 250 to 500°C at a constant strain rate of 0.1 s⁻¹; a) at 250, b) at 300, c) at 400, and d) at 500°C.
Figure 5.17 Optical micrographs in Al$_2$O$_3$/6061Al composite deformed at 300°C at strain rates of a) 1 and b) 0.01 s$^{-1}$. 
Figure 5.18 Microstructures in 6061Al alloy deformed in a temperature range of 200 to 500°C at a constant strain rate of 0.1 s⁻¹; a) at 200°C, b) at 300°C, c) at 400°C, and d) at 500°C.
5-2-6 Microstructural Development in the Composite During Hot Compression

Microstructures of deformed samples were studied using both optical and electron microscopies. The effect of deformation temperature and strain rate on microstructures was investigated. Deformed structures in different pre-treatment conditions were also observed.

5-2-6-1 Optical Microscopy

Figures 5.16(a-d) show a group of optical micrographs of Al₂O₃/6061Al alloy deformed in a temperature range from 250 to 500°C at a strain rate of 0.1s⁻¹. Heavily elongated grains give structure a streak look in samples deformed at 250 °C, and initial grain boundaries can hardly be seen because of shearing. Fine banded structure is present in the sample deformed at this low temperature. As deformation temperature increases above 300°C, the bands disappear and elongated grains with identifiable boundaries are displayed (Fig.5.16b-d).

Microstructures in the composite deformed at 300 °C in strain rates of 1 and 0.01 s⁻¹ are shown in Fig.5.17a and b. At strain rate of 1 s⁻¹, the sample displays fine elongated grains with distorted and banded boundaries. At low strain rate, the grains are elongated but not heavily sheared and distorted. Microstructures of 6061Al monolithic alloy deformed at a strain rate of 0.1s⁻¹ in the temperature range 200 - 500°C are shown in Fig. 5.18(a-d). Shearing or substructure bands can be seen in the samples deformed at 200 and 300°C. The samples deformed at 400 and 500°C exhibit the structure with elongated grains (Fig.5.18d).
Figure 5.19 TEM micrographs in Al₂O₃/6061Al composite deformed at temperatures, a) 200, b) 300, c) 350, d) 400, e) 450, f) 500°C.
Figure 5.20 TEM microstructures in Al$_2$O$_3$/6061Al composite deformed at 300°C at strain rates, a) 1, b) 0.1, c) 0.01, and d) 0.001 s$^{-1}$. 
Figure 5.21 TEM microstructures in Al$_2$O$_3$/6061Al composite deformed at 400°C at strain rates, a) 1, b) 0.1, c) 0.01, and d) 0.001 s$^{-1}$. 
5-2-6-2 Transmission Electron Microscopy

Figures 5.19(a-f) are a series of transmission electron micrographs showing microstructural development during deformation at the temperature between 200 and 500°C at strain rate of 0.1s⁻¹. The specimen deformed at 200°C reveals a dynamically recovered substructure which has a distinct cellular character with a high density of dislocations at the cell walls and within the cells. The subgrain boundaries become more sharply defined and subgrain size becomes larger as the deformation temperature increases (at 300 and 350°C, Fig.5.19b-c). A coarser subgrain structure is found in the samples deformed above 400°C (Fig.5.19d-f). The dislocation density in samples deformed at 450 and 500°C is still quite high, probably due to CTE mismatch (coefficient of thermal expansion) during quenching from the high temperature.

Figures 5.20 and 5.21 display two groups of TEM micrographs taken from the composite deformed at 300 and 400°C, respectively. Each group has four micrographs that are in sequence of the specimens deformed at strain rates of 1 to 0.001s⁻¹. At 300°C, small equiaxed cell structure with ill-defined boundaries is presented and dislocation density is high in the sample deformed at a strain rate of 1s⁻¹ (Fig.5.20a). As the strain rate decreases, the boundaries of the substructure become sharper and the subgrains become larger (Fig.5.20b-d). The subgrains are larger in the specimens deformed at 400°C compared to the specimens deformed at 300°C (Fig.5.21). Subgrain boundaries are well defined in the samples deformed at all strain rates tested. Cellular structure with dense dislocations can be found within the subgrains in the specimens deformed at strain rates 0.1 to 1s⁻¹ as shown in Fig.5.21(a,b). At low strain rates (0.01 and 0.001s⁻¹), the samples exhibit well developed large subgrains with the high density of dislocation networks.
Table 5.2 Average subgrain size (μm) in deformed $\text{Al}_2\text{O}_3$/6061Al composite (and 6061Al alloy) at different temperatures and strain rates.

<table>
<thead>
<tr>
<th>Strain Rate</th>
<th>250°C</th>
<th>300°C</th>
<th>350°C</th>
<th>400°C</th>
<th>450°C</th>
<th>500°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 s$^{-1}$</td>
<td>0.86</td>
<td>1.5</td>
<td>1.56</td>
<td>2.17</td>
<td>3.01</td>
<td>4.05</td>
</tr>
<tr>
<td></td>
<td>(1.4)</td>
<td>(1.98)</td>
<td>(2.3)</td>
<td>(3.52)</td>
<td>(4.2)</td>
<td>(4.84)</td>
</tr>
<tr>
<td>0.1s$^{-1}$</td>
<td>1.15</td>
<td>1.94</td>
<td>2.27</td>
<td>3.07</td>
<td>3.77</td>
<td>5.77</td>
</tr>
<tr>
<td></td>
<td>(1.8)</td>
<td>(2.83)</td>
<td>(3.1)</td>
<td>(4.51)</td>
<td>(6.0)</td>
<td>(7.8)</td>
</tr>
<tr>
<td>0.01s$^{-1}$</td>
<td>1.4</td>
<td>2.20</td>
<td>2.5</td>
<td>3.5</td>
<td>4.66</td>
<td>6.1</td>
</tr>
<tr>
<td></td>
<td>(2.2)</td>
<td>(3.4)</td>
<td>(3.95)</td>
<td>(5.92)</td>
<td>(6.7)</td>
<td>(8.3)</td>
</tr>
<tr>
<td>0.001s$^{-1}$</td>
<td>1.87</td>
<td>2.36</td>
<td>2.8</td>
<td>4.8</td>
<td>6.04</td>
<td>7.1</td>
</tr>
<tr>
<td></td>
<td>(2.8)</td>
<td>(4.47)</td>
<td>(5.0)</td>
<td>(6.62)</td>
<td>(8.53)</td>
<td>(10.53)</td>
</tr>
</tbody>
</table>
Figure 5.22 Plots of inverse of subgrain diameter verses longitude of the temperature compensated strain rate, $Z$, for both $\text{Al}_2\text{O}_3/6061\text{Al}$ composite and 6061Al alloy.
Figure 5.23 Microstructures in Al₂O₃/6061Al composite with T4 condition deformed at temperatures, a) 250, b) 300, c) 350, and d) 450°C at a constant strain rate of 0.1 s⁻¹.
Subgrain size in deformed $\text{Al}_2\text{O}_3/\text{6061Al}$ composite and 6061Al alloy at different temperatures and strain rates are listed in Table 5.2. The results show that the subgrains became larger as the deformation temperature increased and strain rate decreased. Effect of strain rate and temperature on subgrain size in both reinforced $\text{Al}_2\text{O}_3/\text{6061Al}$ and unreinforced 6061Al alloys are shown in Fig.5.22a and b respectively. The data are scattered, but there is a tendency that $d^{-1}$ increases with $\ln Z$ for both reinforced and unreinforced materials that indicates that subgrain size is related to deformation temperature and strain rate in these two alloys.

5-2-6-3 Effect of Pre-Treatment Conditions on Deformed Structure of the Composite

Optical microstructures of deformed composite in T4 condition are shown in Fig.5.23(a-d). It can be seen that the grains are severely sheared in the samples deformed at 250°C. As the temperature increases to 350°C, elongated grains are identifiable. No equiaxed grains can be found in these samples indicating that the material does not undergo dynamic recrystallization in the present conditions.

Figures 5.24(a - d) are a group of TEM micrographs taken from samples of the composite hot compressed at 0.1 s$^{-1}$ between 200-500°C in T4 condition. Substructure in the specimen deformed at 200°C was not well developed. Fine cell structure is present in the massive dislocation clouds as shown in Fig.5.24a. At 300°C, some sub-boundaries can be found, but the structure is still full of dislocation clouds. As the temperature increases further, subgrain boundaries become sharper and dislocation density decreases as shown in Fig.5.24(c-d). Needle and rod-shaped precipitates can be observed in the samples deformed at 300 and 400°C. The microstructure in the sample deformed at 500°C is free of the rod-shaped precipitates and well defined subgrain can be seen.
Figure 5.24 TEM micrographs in Al₂O₃/6061 Al composite in T4 condition deformed at temperatures, a) 200, b) 300, c) 400, d) 500°C at a strain rate of 0.1 s⁻¹.
Figure 5.25 TEM micrographs in Al₂O₃/6061Al composite in T4 condition deformed at 400°C in a strain rate range of 1 to 0.001 s⁻¹; a) at 1s⁻¹, b) at 0.1s⁻¹, c) at 0.01s⁻¹, and d) at 0.001s⁻¹.
Figure 5.26 Annealed Structures of Al5O3/6061Al alloy deformed at: a) 200, b) 300, c) 400, and d) 450°C and at a strain rate of 0.1s⁻¹ followed by annealing at 500 °C for an hour.
Figure 5.27 Static recrystallized grains in hot deformed composite at 400°C and strain rate of 1 to 0.001s⁻¹.
Substructures in the samples deformed at 400°C in a strain rate range of 1 to 0.001 s\(^{-1}\) are shown in Fig.5.25(a-d). It can be seen that subgrain boundaries are distinguished at this temperature, even at strain rate of 1s\(^{-1}\). High density of dislocations and undeveloped cell structure are found within the subgrains in the samples deformed at strain rates of 1, 0.1, and even 0.01s\(^{-1}\). Rod-shaped precipitates are clearly seen in slowly strained samples in Fig.5.25(c-d).

5-2-7 Static Recrystallization During Annealing in Hot Worked Composite

Hot worked reinforced and unreinforced samples were annealed at 500°C for one hour. The optical micrographs of annealed composite samples deformed in temperature range of 200 to 450°C at strain rate of 0.1 s\(^{-1}\) are shown in Fig.5.26(a-d). It can be seen that the sample deformed at 200°C exhibits equiaxed fine grain structure. As deformation temperature increases, the grain size increases following annealing. At 400°C, the composite displays a structure with specially coarse grains (Fig.5.26e) but the sample deformed at 450°C exhibits fine elongated grains after annealing for an hour at 500°C. It is found that the structures of the samples deformed at 450 and 500°C followed by annealing are quite similar to that of un-annealed sample. The micrographs of the samples deformed at 400°C and strain rates of 1 to 0.001s\(^{-1}\) are shown in Fig.5.27 (a-d). The samples deformed at high strain rates (1 & 0.1 s\(^{-1}\)) exhibit large grain structure whilst samples deformed at strain rates of 0.01 and 0.001 display an unrecrystallized structure.

Grain size distribution in hot worked composite after annealing is shown in Fig.5.28. The figure displays a three dimension diagram which shows effect of deformation temperature and strain rate on static recrystallized grain structure of the reinforced
Figure 5.28 Grain size distribution of the composite deformed at 250 to 500°C and at strain rates of 1 to 0.001 s\(^{-1}\).

Figure 5.29 Grain size distribution of 6061Al alloy deformed at temperatures and strain rates tested.
Table 5.3 Recrystallized grain sizes (μm) in Al₂O₃/6061Al composite (and 6061Al alloy) deformed at different temperatures and strain rates followed by annealing at 500°C for an hour.

<table>
<thead>
<tr>
<th></th>
<th>200°C</th>
<th>250°C</th>
<th>300°C</th>
<th>350°C</th>
<th>400°C</th>
<th>450°C</th>
<th>500°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 s⁻¹</td>
<td>30</td>
<td>57</td>
<td>70</td>
<td>240</td>
<td>305</td>
<td>N*</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>(110)</td>
<td>(167)</td>
<td>(680)</td>
<td>(660)</td>
<td>(N)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.1 s⁻¹</td>
<td>20</td>
<td>30</td>
<td>80</td>
<td>250</td>
<td>295</td>
<td>N</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>(49)</td>
<td>(160)</td>
<td>(216)</td>
<td>(980)</td>
<td>(N)</td>
<td>(N)</td>
<td>(N)</td>
</tr>
<tr>
<td>0.01 s⁻¹</td>
<td>50</td>
<td>110</td>
<td>300</td>
<td>N</td>
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<td>N</td>
<td>N</td>
</tr>
<tr>
<td></td>
<td>(260)</td>
<td>(590)</td>
<td>(N)</td>
<td>(N)</td>
<td>(N)</td>
<td>(N)</td>
<td>(N)</td>
</tr>
<tr>
<td>0.001 s⁻¹</td>
<td>80</td>
<td>200</td>
<td>550</td>
<td>N</td>
<td>N</td>
<td>N</td>
<td>N</td>
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<td>(300)</td>
<td>(886)</td>
<td>(N)</td>
<td>(N)</td>
<td>(N)</td>
<td>(N)</td>
<td>(N)</td>
</tr>
</tbody>
</table>

* N - non-recrystallized
alloy. It shows that recrystallized grain size increases as strain rate decreases and
temperature increases. In high temperature and low strain rate region, structure is
found not change after annealing. A similar distribution of the grain size is also found
in the monolithic alloy (Fig.5.29). The measurements of the grain size for the two
materials are listed in Table 5.3.

5-3 Discussion

5-3-1 Flow Behaviour Dependent on Strain Rate and Temperature in
Al₂O₃ Particle Reinforced 6061Al Alloy

The results show that the flow stress rise to a maximum value and then approaches a
steady-state at all testing temperatures except at 200°C. The curve at 200°C is
characterised by strain hardening in whole range of strain. The flat flow curves at high
temperature were resulted from balance between strain hardening and dynamic
softening. They are typical of the form expected when dynamic recovery occurs,
which is balance between generation and rearrangement or annihilation of dislocations
during deformation (150,152).

The flow curves of the composite and the monolithic alloy are similar in the shape
indicating that the composite displays a similar deformation behaviour as the
monolithic alloy. The flow stresses are independent on strain but dependent on strain
rate and deformation temperature above 250°C. Effect of strain rate on flow behaviour
is reflected by strain rate sensitivity, m, or stress exponent n'. The results show that
the composite has similar m values as the monolithic alloy in whole temperature range
tested. The n' values for the two materials are also similar although the value of n' for
the composite is slightly higher. High exponent for 6061Al composites has been
Figure 5.30 The flow stresses as a function of deformation temperatures for both reinforced and unreinforced alloys at the strain rates; a) 1, b) 0.1, c) 0.01, and d) 0.001s⁻¹.
reported by several researchers (220,224,226). The reason for this was considered as the presence of a threshold stress in the composites during high temperature deformation (227,231). Although the precise mechanism responsible for the existence of a threshold stress has not yet been identified, it has been considered to be caused by dislocation pinning in ODS alloy, and could be enhanced by load transfer between reinforcement and matrix, or work hardening in the composites (227,228,231). However, the present results do not show a particular high n' value for the composite as reported by other workers (220,226,227,231). The possible explanation for this is that the size of the particles in the present material are too large (25μm in the present compared to < 5 μm in others studies). These large particles are not only unable to pin the dislocations but also have low strengthening ability (25).

Effect of the temperature on the flow behaviour can be estimated by evaluation of activation energy for deformation, Q. In the present study, Q value for the composite is higher, 222 kJ/mole, than 204 kJ/mole for the monolithic alloy. High activation energy for reinforced materials has been reported, and it is considered as a result of rapidly drop of the flow stress as the temperature increases (224). This can be seen from Fig.5.30 which shows flow stresses as a function of deformation temperature at identical strain rate. The difference in flow stresses between the two materials is larger at low temperature. As deformation temperature increases, the difference in the flow stresses decreases. At 500°C, the difference in flow stresses for the two materials is quite small. This behaviour could be due to transition in the structure and properties described by Humphreys (209, 210). It has been found that transition in the microstructure and mechanical properties could occur in the alloys containing particles at certain high temperature. The transition was considered as a result of higher diffusivity and faster climb of the dislocations at the temperatures which is higher than a critical temperature. According to transition theory, accumulate rate of dislocations
around particles is higher than the rate of removing from the particles during deformation at low temperature. This will cause a high work hardening rate in the alloy. As temperature increases, cross slip and climb of the dislocations is faster and annihilation of dislocation tends to be faster than accumulation. However, no sharp transition in the mechanical properties has been found in the present material and deformation condition because no rapidly changing in flow stress and proof stress has been observed. The rate of decrease in the stress was higher in the composite than in the monolithic alloy as the temperature increased but changing was conducted smoothly. Therefore, it seems that changing in the dislocation structure is a continuing process in the present conditions. Nevertheless, the faster decrease in yield stress and flow stress in the composite could be caused by more than one reasons. In addition of transition on the dislocation structure, decrease in density of dislocations generated by CTE effect can also result in loss in strength of the reinforced alloy at high temperature. This will be discussed in the next section.

5-3-2 Strengthening of the Composite at Elevated Temperature

Strengthening of the composite is attributable to microstructural changes in the matrix caused by adding large amount of reinforcement (8,24,25). These changes include increase in dislocation density and refinement of subgrains and grains. Especially, interaction between dislocations and reinforcement is considered as one of most important strengthening mechanisms (8,24-34). At low temperature, the composite exhibits a high yield stress and flow stress. The Al$_2$O$_3$ particles act as dislocation barriers during deformation and confer a substantial improvement in the strength of the composite over the unreinforced alloy. The flow stress of the composite is higher by 25 - 30 MPa than that of matrix alloy at 250°C. The yield stress or proof stress for the composite is also much higher than that for the matrix alloy at 250°C. However, as the
temperature increases, difference in the flow stresses between the two materials decreases. It can be seen that the flow stress dropped faster for the composite compared to that for the monolithic alloy.

Several authors have discussed the substantial drop observed in the flow stress and yield stress in two phase alloys on increasing the temperature (201,214-217,232,233). Most investigations of the high temperature strength of the two phase alloys have been concentrated on the alloy containing dispersoid particles (201,214-217). Those materials are strengthened owing to Orowan loops at low temperature. As temperature increases, the strength drops rapidly due to dislocation by passing, and to particle coarsening or deformation (201,215,216). However, this would not be applied to the composites that containing very large non-deformable particles. Dislocation structure in the composite is more complicated than Orowan loops.

There are two sources for generation of dislocation in the composites. One is CTE effect, that is, difference in coefficient of thermal expansion will generate dislocations during cooling after thermomechanical processes (43,44). Those dislocations existed previous deformation will interact with hard particles during deformation. Since the particles will stop the movement of the dislocation, deformation resistance increases. This interaction between dislocation and particles will increase yield strength of the composite in the moment of deformation. This effect is quite similar to the effect of grain boundary which acts as barrier of dislocations, but it is much stronger because particles are non-deformable.

Another resource for the composite gaining high density of dislocations is geometrical mismatch between the matrix and particle during deformation (49,50). Mismatch between deformed matrix and non-deformable particle will generate geometrical
necessary dislocations (50,233). Interactions between the dislocation and particle, and
tangle among the dislocations could increase resistance of yielding and initial strain
hardening rate (233).

Both dislocations generated by CTE and geometrically necessary arrays will contributed to strengthening of the composite at low temperature. However, as the temperature increases, those dislocation structures and interaction between those structures will change. Firstly, CTE effect will be reduced since the strains caused by mismatch of CTE will be reduced as temperature increases. Dislocation and internal stresses will be released. In this case, the advantage in the strengthening by high density of dislocations in the composite by CTE effect will be reduced and finally disappear. This could be one of the reasons that yield or proof stresses decrease more rapidly for the composite than for the matrix alloy as temperature increases.

After the material is yield, geometrically necessary arrays will be generated in the matrix around the particles as described by Ashby (50,232). Tangle of those dislocations will make further deformation difficult and the alloy is strain hardened. Strain hardening rate for the composite is much higher than that for the matrix alloy. Even at high temperature (500°C), the composite still displayed a higher strain hardening rate compared to the monolithic alloy. This suggests that effect of the large particle on mechanical behaviour did not disappear at high temperature. Although diffusivity and mobility of dislocations increase at high temperature, addition of geometrically necessary arrays caused by misfit between the particle and the matrix at the beginning of plastic deformation is still available and capable to resist straining.

Grain size could affect strength of the materials and this has been discussed by many researchers based on Hall-Petch equation (234-236). Yield stress of the alloy is
Figure 5.31 Plot of the flow stress, $\sigma$ vs inverse of subgrain size, $d^{-1}$ for the composite.
proportionally related to inverse of square root of grain size. The finer grain size, the strength of the alloys is higher. Initial grain size in the composite is about 21\(\mu\text{m}\) in diameter compared to 105 \(\mu\text{m}\) for the monolithic alloy in the present materials. This fine grain size could further increase yield stress in the composite. However, effect of initial grain size on strength of the materials will decreases as the temperature increases due to the increasing number of deformation modes which become available as the temperature increases (234,237). Thus, initial grain size has no effect on the flow stress if both grain boundary strengthening and grain boundary sliding are not concerned. However, the subgrains formed during deformation could influence strength of the materials. It has been found that the flow stress or yield stress could be inversely related to substructure size during high temperature deformation (151,201). The relationship between steady state flow stress and subgrain size in present conditions can be written as following equation for the composite according to regression of the data in Fig.5.31.

\[
\sigma_c = -5.53 + 167.88 \text{de}^{-1} \tag{5-4}
\]

This linear relationship is in agreement with other studies in aluminium alloys and composites (206,238). The flow stress depends on the size of subgrains and remains constant throughout steady state deformation, because the subgrain size and the dislocation density remain constant (239). It is obvious that the flow stress is higher as the subgrain size is finer. From Table 5.2, it can be seen that subgrain size is much finer in the composite than in the monolithic alloy at identical temperature and strain rate. This could contribute to high flow stress for the composite at high temperature.
Figure 5.32 Deformed structure of the composite at 500°C and at a strain rate of 0.001s⁻¹. Diffraction pattern (SADP) taken from the boundary between grains A and B indicates that the grain boundary is a small angle boundary.
Microstructural Development in the Composite During Hot Deformation

The form of true stress true strain curves indicates that dynamic restoration during hot compression above 250°C is dominated by dynamic recovery. This is supported by microstructure observation in deformed samples. In the optical micrographs, the structures in the samples deformed at temperature below 300°C are characterized by elongated grains containing shear bands. Initial grain boundaries are hard to be identified because of heavy distortion and shearing. The samples deformed above 350°C exhibit elongated grain structure, but shearing bands can not be observed in these samples. This indicates that recovery was more significant at high temperature. The micrographs show that no recrystallization occurred during hot compression in the present deformation condition, even a few small equiaxed grains adjacent to large particles can be found in the samples deformed at temperatures above 300°C.

TEM micrographs show that the samples deformed at 200 and 250°C exhibit fine substructure with identifiable sub-boundaries and high density of dislocations within the subgrains. As the temperature increases and strain rate decreases, subgrains become larger, sub-boundaries become sharper. The subgrain could reach about 8 μm in diameter in the sample deformed at 500°C. The triple point grain boundaries are quite similar to recrystallized large angle boundaries. However, from selected area diffraction pattern (SADP), it was found that most boundaries were low angle boundaries suggesting that these large grains are well developed subgrains instead of recrystallized grains. Figure 5.32 shows the grain structure in the sample deformed at 500°C with strain rate of 0.001 s⁻¹. Selected area diffraction patterns indicate that the boundaries between grains A and B, and C and D are small angle boundaries but the boundaries between grains A, B and C, D are large angle boundaries. These large
angle boundaries are considered to be initial grain boundaries prior to deformation, which enclose at least two subgrains separated by low angle boundaries after deformation. The area enclosed by large angle boundaries is elongated because of the deformation. These microstructural observations indicate that the deformation can be considered to be characterized by dynamic recovery in the reinforced material at high temperatures.

As mentioned above, subgrain size is dependent on strain rate and temperature of deformation but independent of strain. The repolygonization mechanism proposed by McQueen et al. (238) stresses that the subboundaries are continually broken up by the flux of dislocations and continually reform at a spacing defined by the rate and temperature of deformation so that no matter how large the strain is, the substructure always appears the same during a steady-state deformation. This means that the subgrain size, $d$, can therefore be related to the temperature compensated strain rate ($Z$). As per Fig.5.22, the relationships between the subgrain size and deformation parameters in the reinforced and the monolithic alloys can be written as:

\[
d_{c}^{-1} = -0.97 + 0.034 \ln Z_c \quad \text{(composite)} \quad (5-5)
\]

and

\[
d_{m}^{-1} = -0.71 + 0.027 \ln Z_m \quad \text{(monolithic alloy)} \quad (5-6)
\]

The equations show that subgrains coarsen as parameter $Z$ increases (that is the temperature increases and strain rate decreases). This is in agreement with observations made by McQueen et al. (238,239). At high temperature and low strain rates, the greater number of thermally activated events per unit strain increases the dislocation mobility leading to greater annihilation, increased dynamic recovery, and a lower steady-state dislocation density (239,240). This can be observed from Fig.5.32.
which shows that large subgrains (up to 8μm in diameter) with low density of dislocations in the sample deformed at 500°C and 0.001 s⁻¹ of strain rate compared to small subgrains (1~2μm in diameter) while the sample was deformed at 250°C (Fig.5.19b). It should be mentioned that the dislocation structure present in these TEM micrographs may be as a result of deformation as well as due to CTE mismatch during quenching from deformation temperature.

The structural study shows that the subgrains are finer in the composite than in the monolithic alloy under identical deformation conditions. This could be resulted from high density of the dislocations. It is known that difference in CTE and geometrically mismatch could produce high density of dislocation in the reinforced alloy. These additional dislocations not only increase resistance of deformation by tangling and interaction with each other but can also reduce distance between the walls and subgrain size by restriction of mobility of the dislocations. The structural details of micrographs in Fig.5.19a,c show that substructures in the area around large particles are finer than that in the area further away from the particles. Evidently high dislocation density in vicinity of the particles seems to be responsible for fine substructure observed.

Another observation can be made from equations 5-5 and 5-6 in which the regression coefficient in equation 5-5 is larger compared to equation 5-6. This indicates that subgrain coarsening in the composite is faster than that in the monolithic alloy as temperature increases or strain rate decreases. This could further support the suggestion that, as temperature increases, the number of dislocations generated by CTE phenomenon and geometrically mismatch will be reduced and the substructure will coarsen and steady state stress will decrease in an additional higher rate compared to that for the monolithic alloy.
5-3-4 Effect of Solution Treatment on Deformation Behaviour and Structural Development

Solute elements in the matrix of the composite pre-treated in OT condition are in a precipitate form since the material is overaged at 500°C following slow cooling. These precipitates do not affect flow behaviour dynamically since such precipitates are relatively stable during deformation of the material at the temperatures below 500°C. However, the alloying elements in the material pre-treated in T4 condition are mostly in the solution, which could dissolve during straining at elevated temperature and influence flow behaviour of the material. This can be seen from flow curves of T4 Al₂O₃/6061Al in Fig.5.9 which displays a flow curve in which the stress first reaches maximum value then decreases as straining continuing. Decrease in flow stress as straining indicates that the alloy was softened dynamically during deformation. There are two possibilities for this dynamic softening. One is occurrence of dynamic recrystallization (151), and another is strain induced (or dynamic) precipitation (162). Microstructural studies indicate that no dynamic recrystallization occurred in the reinforced alloy in the present deformation conditions. The dynamic softening observed is thus a result of strain induced precipitation and coarsening. At high temperature, alloying elements Mg and Si which are mostly in solution prior to deformation will precipitate and fine particles precipitated during holding prior to deformation will coarsen due to straining. These two processes will lead the material strain softening.

It can be seen from Fig.5.9a that at 200°C, the flow curve exhibits continuing strain hardening but strain hardening rate is lower compared to that of the material in OT condition (Fig.5.1b). Strain softening occurs in the samples deformed at 250 to 400°C, and the flow stresses become steady-state at the temperatures above 450°C.
Figure 5.33 Plot of $\ln(\sinh(\alpha\sigma))$ vs $1/T$ for the composite in both OT and T4 conditions.

Figure 5.34 Comparison of subgrain size of the composite in OT and T4 conditions.
These observations indicate that, at 200°C, T4 Al_2O_3/6061Al is much stronger than OT Al_2O_3/6061Al because of solution strengthening but strain induced precipitation reduces strain hardening rate in T4 material during deformation. As temperature increases to 250°C, precipitation and coarsening are more sufficient leading to obvious strain softening. It has been found that at the temperature above 250°C, alloying elements could precipitate to β' phase which made the material soft (Fig.3.3). Straining could accelerate formation of β' phase and decrease flow stress dynamically. This flow behaviour of T4 material could be seen until the deformation temperature reached 450°C at which the flow stress is in a steady-state and dynamic changing is hardly to be observed. This indicates that either dissolution was limited or precipitates coarsened and stabilized during holding prior to deformation, which would not affect flow stress dynamically. TEM micrograph (Fig.5.24d) shows that no rod-shaped β' precipitates present in T4 alloy deformed at 500°C indicating that absence of strain softening was caused by insufficient dissolution at such high temperature.

The effect of strain rate on the maximum flow stress of the samples in T4 condition is not very different from that of the samples in OT condition since the m values in the both conditions are quite similar at 400°C. Although the precipitates are coarser as the strain rate decreases (Fig.5.25), it may not affect the maximum flow stress at the beginning of straining since the samples possess similar structure prior to deformation. However, the effect of the temperature on the maximum flow stress was significant. This can be seen from the plot of ln[sinh(ασ)] vs 1/T in Fig.5.33. A non-linear relationship of ln[sinh(ασ)] vs 1/T is present in T4 alloy compared to a linear relationship for the alloy in OT condition. The decrease in flow stress with the temperature was faster at the temperature below 350°C than above 350°C. It seems that at low temperature (200 and 250°C), most alloying elements (Mg and Si) were in solution or precipitated to fine precipitates during holding at the deformation
temperature prior to compression leading to a high flow stress. As temperature increased, the alloying elements would precipitate to $\beta'$ or Mg$_2$Si phases after holding at the deformation temperature prior to straining resulting in a sharp decrease in the maximum flow stress. However, at the temperature above 400°C, dissolution was limited and the maximum flow stresses would not decrease so fast as in low temperature range (Fig.5.33). Evidently, there could be two activation energies for deformation of T4 alloy; high value in temperature range between 250 to 350°C (great slope of the plot ln[sinh($\alpha\sigma$)] vs 1/T), and low value between 400 to 500°C, which was similar to that for OT alloy. As discussed above, the high activation energy in T4 alloy is attributed to strain induced precipitation and its reaction with dislocation during deformation.

Substructural development in the Al$_2$O$_3$/6061Al in T4 condition was sluggish. This can be seen from Fig.5.24(a-d) which show that the cell structure is not well developed at the temperatures until 300°C. Fine and dense dislocation networks are presented in the specimens deformed at the temperatures below 400°C. Comparison of the substructure size in T4 Al$_2$O$_3$/6061Al and OT Al$_2$O$_3$/6061Al is shown in Fig.5.34 (It is hard to measure the subgrain size in T4 alloy deformed at 250°C because of its less recovered substructure). Substructure is finer in T4 alloy than in OT alloy, especially in those samples deformed at temperatures below 400°C. The fine substructure is caused by pinning effect of solute and initial precipitates present in T4 material, especially solute pinning at low temperature delays the development of the substructure. At the temperature above 450°C, movement of the solute atom is accelerated, and pinning effect is reduced which leads to a similar substructure as that in the material in OT condition.
It should be mentioned that precipitates interacted with dislocations only at their initial stage (β" and β' phases). Over-aged equilibrium phase Mg₂Si has no pinning ability. This is why the rod-shaped precipitates present within subgrain and on the boundaries in the samples deformed above 400°C (Fig. 5.24 and 5.25) have shown no pinning effect on substructure.

5-3-5 Static Recrystallization and Grain Structure in Hot Worked Composite

Recrystallization of deformed structure is driven by stored energy introduced by deformation (112). Alloy deformed at low temperature has high stored energy but as deformation temperature increases, dynamic restoration occurs which will reduce the stored energy. This may affect microstructural development in the following thermomechanical process of the material. The composite samples deformed at the temperatures between 200 and 500°C have shown recovered substructures. Annealing at 500°C may lead to recrystallization in these hot worked samples. Since degree of recovery in these samples is varied, the recrystallized structure will be different. The results show that the samples deformed at low temperature (200-250°C) recrystallized to fine grained structure. As deformation temperature increases, the recrystallized grain size becomes larger. It seems that there is a certain relationship between recrystallized structure and hot deformed structure prior to annealing. The sample deformed at low temperatures contained less developed substructure with high density of dislocations. During annealing, the highly dislocated substructure could generate more nuclei, and recrystallized to fine grained structure. On the other hand, the samples compressed at higher temperature (300-400°C) have well recovered substructure with low density of dislocations. Such a substructure would offer a low nucleation rate thus would result in a few large grains during annealing. However, the samples deformed above 450°C
Figure 5.35 Substructures in Al$_2$O$_3$/6061 Al material deformed at 450°C using a strain rate of 0.001 s$^{-1}$ followed by annealing at 500°C for one hour. Selected area diffraction patterns (SADP) obtained from grains A, B and grain boundary A & B are similar. The patterns are indexed with the zone axis [011]. Negligible misorientation between grains A and B indicates that these grains are subgrains.
Figure 5.36 The critical temperature for static recrystallization in deformed materials during annealing; a) the composite and b) the monolithic alloy.
and at strain rates under 1s⁻¹ show similar structures after being annealed as that before annealed. The TEM micrograph in Fig.5.35 shows a structure in the sample deformed at 450°C and at a strain rate of 0.001s⁻¹ followed by annealing. Diffraction patterns show that the grain structure in this annealed sample consists of subgrains with small angle boundaries. This indicates that recovered substructure in the sample deformed at such high temperature was so stable that not enough stored energy was left to initiate recrystallization during following annealing.

The deformation temperatures at which the samples show no recrystallization during annealing are plotted against strain rate as shown in Fig.5.36(a,b) for Al₂O₃/6061Al and 6061Al alloys respectively. The line in the figure represents a critical temperature at various strain rates. If the deformation is conducted below this critical temperature, samples will recrystallize after annealing. Above this temperature, samples will not undergo static recrystallization during following annealing. The critical temperature decreases as the strain rate decreases. The reason for this is that as strain rate decreases, there will be more time for annihilation of dislocations and the deformed structure can be recovered in a higher level at lower temperature. This behaviour is quite similar to so called transition mentioned above. However, this transition in structure is not due to presence of large particles in the alloy because such a behaviour has also been found in the monolithic alloy, and the transition was found to be at same temperatures and strain rates as exhibited by the composite. These observations lead to believe that transition of dislocation structure during hot working seems mainly dependent on alloying element and thermal activation of the alloy rather than due to presence of large ceramic particles.

It can be noticed that large ceramic particles did not hinder grain boundary movement effectively in the reinforced material deformed below critical temperature. Even the
grain size is smaller in the composite compared to the monolithic alloy deformed under identical condition, the grain size is still too coarse in the composite. This indicates that large reinforcement can only restrict grain growth in certain conditions, and fine grain structure in reinforced material seems mainly due to high nucleation rate caused by stimulation of the large particles. The sample deformed at 200°C exhibited a structure similar to cold deformed structure which was almost unrecovered. During annealing, large particles would act as recrystallization sites and thus enhance nucleation rate. As deformation temperature increased, the high density of geometric necessary dislocations was removed by climb resulting from dynamic recovery and in such a situation, there would be no advantage for the large particles to stimulate nucleation. Low nucleation rate leads to large recrystallized grains.

The results show that recrystallized grain size after hot working is dependent on both deformation temperature and strain rate and there are three regimes of grain size. Fine grains in low temperature and high strain rate region; coarse grains in intermediate temperature and low strain rate region; and unrecrystallized substructure in high temperature and low strain rate region. Such an information could be useful in material processing for control of the structure during hot working and subsequent heat-treatment in metals and alloys containing hard particles.

5-4 Summary

1. Al₂O₃ particle reinforced 6061Al alloy has higher strength at elevated temperature compared to the monolithic alloy. The composite exhibits high yield stress and strain hardening rate in whole range of temperatures and strain rates studied. However, the composite is much stronger than the matrix alloy at low temperature but as temperature increases, the difference in strength between the two materials decreases, that is, the
strength of the composite tends to be similar to that of the monolithic alloy. This is caused by annihilation of dislocations and decrease in CTE effect in the composite at high temperature.

2. Dynamic recovery is a principal restoration process in both reinforced and unreinforced 6061Al alloys during high temperature deformation. Substructure is developed from cell structure with high density of dislocations at 250°C to well developed subgrains with sharp boundaries at 500°C. Subgrain size is dependent on deformation temperature and strain rate.

3. The reinforced material displays finer substructure than the monolithic alloy at whole temperature and strain rate range tested. Refinement of the substructure in the reinforced material is more obvious at low temperature and high strain rate. As temperature increases and strain rate decreases, coarsening of the substructure is slightly faster in the reinforced material than that in the monolithic alloy. This is caused by decrease in dislocation density resulted from reduce in the CTE effect and faster removal of the dislocations around the particles at high temperature.

4. Strain induced precipitation results in dynamic softening of the materials in T4 condition, which leads to a peak shaped flow curve. This phenomenon is more obvious while the alloys are deformed in a temperature range of 250 to 400°C. At above 450°C, no dynamic softening occurs because of absence of strain induced precipitation at this high temperature.

5. Precipitates, Mg$_2$Si, have shown effect on the flow behaviour and microstructural development only in their initial stage - β" zone. These zones as well as solute atoms react with dislocations and sub-boundaries at low temperatures to produce fine
substructure in the material pre-treated in T4 condition. Rod-shaped or equilibrium Mg$_2$Si phases at high temperature are found no much effect on deformation behaviour and substructure in Al$_2$O$_3$ reinforced 6061Al alloy since they have no pinning ability.

6. Activation energy for deformation, $Q$, is 222 kJ/mole for the composite. It is higher than 204 kJ/mole for the monolithic alloy in a temperature range of 250 to 500°C and strain rates of 0.001 to 1s$^{-1}$. This indicates that the flow stresses of the composite are more dependent on deformation temperature, that is, strength of the reinforced alloy drops more rapidly as the temperature increases. Deformation of T4 material can not be described by a single value of activation energy due to precipitation of alloy elements Si and Mg in solution prior to or during deformation at high temperature.

7. Static recrystallization only occurs in hot worked materials which are deformed in a certain low temperature region during subsequent annealing. It is found that as deformation temperature increased to a critical point, no recrystallization occurs and well recovered substructure is remained after annealing. The critical deformation temperature is dependent on strain rate. As strain rate decreases, critical temperature decreases. Both reinforced and unreinforced materials show same critical temperatures.

8. There are two regions for the distribution of recrystallized grain size depending on deformation temperature and strain rate. Fine grain size in low temperature and high strain rate region; and coarse grains in high temperature and low strain rate region. The sample deformed at 200 °C exhibits fine recrystallized grains in a size similar to cold worked sample after annealing. As deformation temperature increases, recrystallized grain size increases. At the temperature near to critical point, grain size can be as large as 600μm in the composite and 1000μm in the monolithic alloy after being annealed.
CHAPTER 6

GENERAL CONCLUSIONS AND SUGGESTIONS FOR FUTURE WORK

In the previous chapters, we have reported on the ageing response, recrystallization and hot working behaviour of Al$_2$O$_3$ particle reinforced 6061 aluminium alloy. In the present chapter, the main conclusion from the preceding sections will be summarized; suggestions will then be made for future work on this material. The work has applicability in understanding the ageing behaviour of particle reinforced 6061 aluminium alloy, microstructural development during ageing, and for establishing proper condition for artificial ageing processes of the material in industry. The studies of particle stimulating nucleation of recrystallization and restricting grain growth in the composite give us an opportunity to further understand reaction between the particle and the matrix, which could prudently influence microstructural development during thermomechanical processes. The work also makes a contribution to the understanding of the hot deformation behaviour of particle reinforced 6061 aluminium alloy from both point of views of mechanical and microstructural development.

I) General Conclusions

The presence of high volume fraction of large particles could affect microstructure, and thermomechanical behaviour of 6061 aluminium alloy. This is because the large particles introduce additional dislocations and internal strains to the material by means of CTE effect and geometric accommodation between the particle and the matrix. This high density of dislocations and internal strains will modify the response of the
material to ageing and recrystallization as well as deformation at elevated temperature. The important features can be summarized as follows:

1. The reinforced materials have a similar age hardening capability as the monolithic alloy. Peak hardness of the materials is attributed to the formation of the $\beta''$ zone which is found to be the only age hardening agent in both reinforced and unreinforced 6061Al alloys in the present ageing conditions. Overage of the alloys is caused by coarsening of the $\beta''$ zone in the temperature range of 125 to 170°C.

2. The presence of large particles increases dislocation density and internal stress in the reinforced materials. This doesn't change ageing sequence but could cause acceleration of formation and coarsening of the $\beta''$ zones and $\beta'$ phase, and thus ageing is accelerated in the composites. Acceleration of ageing has only been found when the alloy is aged at certain high temperature (170°C at the present). When the temperature is low (125°C), formation and coarsening of the $\beta''$ phase is considerably slow and acceleration of ageing is not obvious in the reinforced materials.

3. DSC analysis shows that introduction of the large particles can decrease the formation temperatures of $\beta''$ zones and $\beta'$ phase suggesting that both formations of $\beta''$ and $\beta'$ are accelerated by high density of dislocations or residual stresses. Even though the $\beta''$ zones nucleate uniformly in the matrix, coarsening of the zones could be accelerated due to high diffusivity conducted by the dislocations and residual stresses in the composites.

4. Deformation prior to ageing can reduce formation of $\beta''$ zones and degrade hardening ability in 6061Al composite during artificial ageing at 125°C. Reduction of $\beta''$ zones is caused by accelerated formation of $\beta'$ phase because dislocation density
introduced by straining is extremely high. Therefore, the peak hardness in deformed material appears much earlier but the value is lower compared to undeformed material.

5. The presence of high volume fraction of large non-deformable Al$_2$O$_3$ particles leads to non-uniform straining in the matrix around the particles resulting in a high density of microbands and geometrical necessary dislocation networks in the reinforced alloy during cold working. These characteristics of deformed structure could affect thermomechanical behaviour and microstructural development in the subsequent processes such as ageing and annealing.

6. High density of dislocations and distortion in the areas around particles can contribute to stimulation of nucleation at the particles. Closely-located particle groups have a strong capability to stimulate nucleation of recrystallization. High nucleation rate caused by large particle stimulation accelerates recrystallization kinetics in the composite thus resulting in a lower recrystallization temperature compared to the monolithic alloy.

7. The reinforced material exhibited finer and more equiaxed recrystallized grains than monolithic 6061Al alloy in fully recrystallized condition. This could be the results of high nucleation rate and restriction of interparticle spacing in the reinforced material. By comparison, reduction and annealing temperature have shown less effect on the grain size in the composite than in the monolithic alloy.

8. Grain growth in the reinforced alloy at high temperature (540°C) is not significant even after 40 hours. The structure showed that grain growth at high temperature was restricted by large particles, that is, abnormal growth was limited within interparticle spacing in reinforced material. By comparison, grain growth in the unreinforced alloy
is obvious, and some large grains are caused by abnormal growth. Inclusions and fine reinforcement particles (about 1 μm) have also shown strong pinning on the grain boundaries in the composite during annealing at 540°C. Insignificant grain growth in the composite is also contributed to the pinning effect of these fine particles.

9. Alloying elements such as Mg and Si have shown strong effect on the mechanical properties in the 6061Al composite. In the forms of either solute in solution or zones from precipitation, the elements increase hardness of the material dramatically due to solute pinning at room temperature. However, at temperature above 250°C, these elements precipitate to coarse second phase and have no pinning effect on dislocations and boundaries. This characteristic of the solute elements results in a high dislocation density and less recovered structure leading to a high nucleation rate in T4 treated materials during annealing. However, as the precipitates become coarse above 250°C, they have little effect on recrystallization kinetics.

10. Dynamic recovery was found to be the only restoration process in reinforced 6061Al alloy during deformation at high temperature. The true stress-true strain curves are the typical form expected when dynamic recovery occurred, that is, the flow stress rises to a maximum value and then approaches a steady-state level above 250°C. Dynamic recovery was also observed from deformed structure which exhibited subgrains with small angle boundaries.

11. Reinforced material has higher strength at elevated temperature compared to the monolithic alloy. The results of hot compression displayed that the composite exhibited high yield stress and strain hardening rate in whole range of temperature and strain rate studied. However, the composite was much stronger than the monolithic alloy at low temperature. As the temperature increased, the difference in compression
strength between reinforced and unreinforced alloys decreased. At 500°C, the composite only showed slightly stronger than the matrix alloy.

12. Activation energy for deformation, Q, is 222 kJ/mole for Al₂O₃/6061Al composite. It is higher than 204 kJ/mole for the monolithic alloy in a temperature range of 250 to 500°C and at strain rates of 0.001 to 1s⁻¹. This indicates that the flow stresses of the composite are more dependent on deformation temperature, that is, strength of the reinforced alloy drops more quickly as the temperature increases.

13. Combined effect of deformation temperature and strain rate on flow behaviour of Al₂O₃/6061Al is not very different from that of monolithic 6061Al alloy suggesting that the reinforced material has similar hot working ability as the unreinforced material in the present deformation conditions. The steady state flow stress is inversely dependent on the subgrain size which is determined by the temperature compensated strain rate.

14. Finer substructure is present in the reinforced material compared to the monolithic alloy deformed in identical conditions, which is attributed to high density of the dislocation caused by geometrical necessary match during deformation. Refinement of the substructure in the reinforced material is found in whole temperature range studied but more obviously at the low temperature and the high strain rate.

15. Alloying elements exhibit considerably effect on high temperature deformation behaviour when they are in the solution. Solute elements Mg and Si in the alloy precipitated in T4 condition induced by straining during deformation at elevated temperature, which resulted in dynamic softening during compression, that is, the flow stress decreases rapidly after reaches the maximum value. Dynamic softening
was found only in low temperature range of 250 to 350°C. Above 450°C, dissolute is limited and strain induced precipitation is not available leading to a steady state flow curve during deformation. The deformation of T4 material can not be described by a single activation energy. A high Q value is obtained at low temperature range and a lower Q value at higher temperatures.

16. The composite in T4 condition exhibits a finer substructure compared to that in OT condition when the material is deformed below 400°C. This is caused by pinning effect of solutes and precipitates during straining. Particularly, solute pinning at low temperature can considerably delay the development of the substructure. At the temperature above 400°C, both solute pinning and precipitate pinning are weakened leading to a similar subgrain size in T4 material as in OT material.

17. Static recrystallization occurs in hot worked composite and matrix alloy during annealing at 500°C. However, there is a critical temperature at which recrystallization is not occurred and deformed substructure remains unchanged after annealing. This critical temperature decreases from 500 to 400°C as the strain rate decreases from 1 to 0.001 s$^{-1}$ for both reinforced and unreinforced materials.

18. Recrystallized grain sizes in hot worked and annealed materials are distributed variously depending on deformation temperatures and strain rates. There are two regions for grain size distribution below the critical temperature; fine grains in a low temperature and high strain rate region and coarse grains in a high temperature and low strain rate region. The sample deformed at 200°C and at a strain rate of 0.1 s$^{-1}$ exhibited a fine grain structure similar to that in cold worked material. However, when it was deformed at 350°C and at a strain rate of 0.001 s$^{-1}$, the sample had grain size as large as 600μm in the composite and 1000μm in the matrix alloy.
II) Suggestions for Future Work

1. It has shown that deformation in the matrix near the particles is non-uniform and nucleation of recrystallization during the following annealing is preferred at large particles. These could produce special deformation and recrystallization texture in reinforced materials. To understand the effect of reduction and annealing temperature on the texture is an important aspect in the research and industrial application of the composite. Furthermore, studies of hot working texture and recrystallization texture in hot worked alloy are also interesting and beneficial.

2. Strength of the Al₂O₃ particle reinforced 6061Al alloy at high temperature has been studied in the present work. It has shown that strength of the composite is much higher compared to the monolithic alloy at low temperature but no much difference in strength at high temperature. Therefore, the reinforced alloy can be deformed at high temperature as easy as the monolithic alloy. However, the presence of high volume fraction of large ceramic particles could affect ductility of the material significantly. To investigate the limited deformation condition in which the materials start failure would be very beneficial to the industry. Future work should involve the effect of strain, strain rate, and deformation temperature on the ductility of the reinforced materials.

3. Annealing or solution treatment following hot working for aluminium alloys are most common processes in industry. These processes could dramatically affect structure of the hot worked materials. The present study showed that both deformation temperature and strain rate could alter grain structure after heat treatment processes. Effect of annealing temperature on grain structure has not been investigated. Large strain and high strain rate may also show influence on recrystallization structure. It will be interesting for both theoretical and industrial purpose to investigate effect of
these parameters in hot working on the structural development during following processes.
APPENDIX 1

In order to plot the data according to Eq.5-2, the value of \( \alpha \) must be known. As first approximation, \( \alpha \) can be taken to be \( \beta/n_1 \) (154,155). \( \beta \) is obtained from

\[
\dot{\varepsilon} = A_2 \exp(\beta \sigma)
\]

(1)

at high stresses, where it is generally independent of temperature; \( n_1 \) is given by

\[
\dot{\varepsilon} = A_3 \sigma^{n_1}
\]

(2)

at low stresses, where it is considered as a temperature-independent constant. \( A_2 \) and \( A_3 \) are constants.

The values of \( \beta \) evaluated from plot of \( \ln \dot{\varepsilon} \) vs \( \sigma \) was found in a range from 0.14 to 0.27 over the temperature range 250 to 500°C for \( \text{Al}_2\text{O}_3/6061\text{Al} \), and 0.17 to 0.28 for 6061Al alloy. The values of \( n_1 \) were obtained from the slope of \( \ln \dot{\varepsilon} \) vs \( \ln \sigma \), and the values decreased from 19 to 6.37 for \( \text{Al}_2\text{O}_3/6061\text{Al} \), and 20 to 5.96 for 6061Al alloy as the temperature increased. Therefore, the values of \( \alpha \) varied from 0.0074 to 0.0423 for the composite, and 0.0084 to 0.048 for 6061Al alloy. These \( \alpha \) values were used to calculate the slopes (\( n' \)) of the best fit lines for each temperature in following equation

\[
n' = \frac{\partial \ln \dot{\varepsilon}}{\partial \ln \sinh(\alpha \sigma)} T
\]

(3)

which was obtained from Eq.5-2 in assuming that the activation energy and the parameter \( A_1 \) were constant over the range of strain rates and temperatures studied (157). 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 S_d decreases as \( \alpha \) increases. However, the largest value of \( \alpha \) calculated as above was still not satisfying to give a minimum S_d. Then the value of \( \alpha \) was extended until smallest S_d was obtained. This was 0.03 for Al_2O_3/6061Al composite, and 0.035 for 6061Al alloy. The corresponding values of stress exponent n' were 5.1 for the composite and 5 for 6061Al alloy. Calculations of \( \alpha \), \( \beta \), and n' are listed in Table A-1 and A-2.

Table A-1 Calculation of \( \ln [\sinh(\alpha \sigma)] \), \( \alpha=0.03 \) for Al_2O_3/6061Al composite

<table>
<thead>
<tr>
<th></th>
<th>250°C</th>
<th>300°C</th>
<th>350°C</th>
<th>400°C</th>
<th>450°C</th>
<th>500°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1s(^{-1})</td>
<td>3.597</td>
<td>2.575</td>
<td>2.002</td>
<td>1.453</td>
<td>0.788</td>
<td>0.3389</td>
</tr>
<tr>
<td>0.1s(^{-1})</td>
<td>3.2365</td>
<td>2.1535</td>
<td>1.5457</td>
<td>0.919</td>
<td>0.4475</td>
<td>-0.0594</td>
</tr>
<tr>
<td>0.01s(^{-1})</td>
<td>2.906</td>
<td>1.6986</td>
<td>1.0474</td>
<td>0.4118</td>
<td>-0.149</td>
<td>-0.5085</td>
</tr>
<tr>
<td>0.001s(^{-1})</td>
<td>2.304</td>
<td>1.2364</td>
<td>0.5872</td>
<td>-0.047</td>
<td>-0.4515</td>
<td>-0.9164</td>
</tr>
<tr>
<td>n''</td>
<td>5.355</td>
<td>5.15</td>
<td>4.85</td>
<td>4.63</td>
<td>5.25</td>
<td>5.46</td>
</tr>
<tr>
<td>r</td>
<td>0.989</td>
<td>0.999</td>
<td>0.999</td>
<td>0.999</td>
<td>0.992</td>
<td>0.999</td>
</tr>
</tbody>
</table>

\( n' = \frac{1}{i} \sum n''_i \), \( i = 6 \), \( n'= 5.1 \), \( S_d = 0.326 \). \( r \) - correlation coefficient
Table A-2 Calculation of $\ln [\sinh(\alpha \sigma)]$, $\alpha = 0.035$ for 6061Al monolithic alloy

<table>
<thead>
<tr>
<th></th>
<th>250</th>
<th>300°C</th>
<th>350°C</th>
<th>400°C</th>
<th>450°C</th>
<th>500°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1s⁻¹</td>
<td>3.437</td>
<td>2.455</td>
<td>1.927</td>
<td>1.32</td>
<td>0.761</td>
<td>0.442</td>
</tr>
<tr>
<td>0.1s⁻¹</td>
<td>2.946</td>
<td>2.103</td>
<td>1.428</td>
<td>0.761</td>
<td>0.314</td>
<td>-0.009</td>
</tr>
<tr>
<td>0.01s⁻¹</td>
<td>2.63</td>
<td>1.606</td>
<td>0.838</td>
<td>0.3573</td>
<td>-0.164</td>
<td>-0.46</td>
</tr>
<tr>
<td>0.001s⁻¹</td>
<td>2.2</td>
<td>1.06</td>
<td>0.442</td>
<td>-0.111</td>
<td>-0.599</td>
<td>-0.93</td>
</tr>
<tr>
<td>$n''$</td>
<td>5.68</td>
<td>4.87</td>
<td>4.54</td>
<td>4.88</td>
<td>5.05</td>
<td>5.04</td>
</tr>
<tr>
<td>$r$</td>
<td>0.997</td>
<td>0.995</td>
<td>0.997</td>
<td>0.998</td>
<td>1.00</td>
<td>1.00</td>
</tr>
</tbody>
</table>

$n' = \frac{1}{i} \sum n''_i, \ i = 6, \ n' = 5, \ \text{Sa} = 0.38$
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