

**CHARACTERIZATION OF INTERMETALLIC PARTICLES IN ALUMINUM  
ALLOY AA5083 SERIES AFTER RECRYSTALIZATION HEAT TREATMENT**

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## ABSTRACT

The distribution and morphology of intermetallic particles' size and distribution after recrystallization heat treatment in aluminium alloy AA5083 series is characterized in this project. Formation of intermetallic particles, Al(Fe,Mn)Si and Mg<sub>2</sub>Si occurred in the matrix during solidification of original cast ingots of rolled sheets [1]. Final size and distributions of these particles in Al-Mg sheets depend on thermo-mechanical processing performed on them. The experimental procedures conducted to obtain the data including metallography, Scanning Electron Microscopy with Energy Dispersive Spectroscopy for specimen's image capture, quantitative measurement technique - Jeffries method. Characterization technique applied in this study has successfully interpreted the intermetallic particles phase distribution as either  $\alpha$  (silicon rich) or  $\beta$  (Fe and Mn rich). The understanding obtained from this study can be best applied in the manufacturer of aluminium alloy in designing required material properties by suitable heat treatment temperatures.

*Keyword:* Intermetallic Particles; AA5083; Recrystallization Heat Treatment

## ABSTRAK

Penyebaran dan morfologi bagi partikel separa logam yang terkandung dalam aloi aluminium siri AA5083 selepas dijalan proses rawatan haba penghabluran semula adalah diklasifikasi dalam projek ini. Pembentukan partikel separa logam, Al(Fe,Mn)Si dan Mg<sub>2</sub>Si terhasil dalam matriks aloi ketika proses solidifikasi bagi gelekkan kepingan logam [1]. Saiz dan penyebaran pada akhirnya bagi partikel tersebut dalam kepingan aloi Aluminium Magnesium adalah bergantung dengan proses thermomekanikal yang dijalankan ke atas kepingan tersebut. Prosedur eksperimen yang dilakukan untuk memperoleh data tersebut adalah metalografi, penggunaan SEM (Scanning Electron Microscopy) dengan EDS (Energy Dispersive Spectroscopy ) bagi pengimejan specimen dan teknik pengukuran kuantitatif – Jeffries method. Teknik pengklasifikasian yang diaplikasi telah berjaya menginterpretasi penyebaran fasa partikel separa logam, samada fasa  $\alpha$  ( kandungan Silikon tinggi) atau fasa  $\beta$  ( kandungan Ferum dan Mangan tinggi) Komposisi element bagi aloi aluminium AA5083, serta partikel separa logam bertitik putih dan gelap dapat dianalisa dengan penggunaan spektra EDS. Hasil kajian ini boleh diaplikasi dalam industri pembuatan aloi aluminium untuk memperoleh spesifikasi yang dikehendaki mengikut kesesuaian pembuatan.

*Kata Kunci:* Intermetallic Particles; AA5083; Recrystallization Heat Treatment

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## **CHAPTER 1**

### **INTRODUCTION**

Aluminium is the most abundant metallic element on the earth and often occurs as alloys, as in combination with other elements such as oxygen and silicon. Nonetheless, aluminium alloys are extremely useful in engineering field.

Pure aluminium has low strength. However, it can be alloyed up to strength of 90 Mpa. Aluminium alloy refers to a complex set of specialty aluminium alloys, where various amounts of other metals are combined with primary aluminium to give strength and specific characteristics for a particular use. It is also often miscalled as scrap aluminium. 5xxx series aluminium alloys are used extensively in the automotive industry as the material for Body In White (BIW). It is a growth market as more aluminium based material is incorporated in cars especially as environmental legislation calls for more efficient vehicles. The use of aluminium alloy instead of steels provides a significant weight reduction advantages.

Being the world second most used metal after steel, there are over 85% of the fabricated aluminium products are rolled as sheets and foil. The understanding of the phenomena of work hardening, recovery, and recrystallization in 5xxx series aluminium alloy is essential not solely to obtain these products but also to control their microstructures to optimize their properties and performance.

## **1.1 Project Objectives**

- i. To implement and validate the characterization technique for the characterization of intermetallic particles in Aluminium Alloy AA5083 after Recrystallization Heat Treatment

## **1.2 Project Scopes**

- i. Wrought Al-Mg alloy AA5083 series will be used in this study.
- ii. Utilization of Scanning Electron Microscope with Energy Dispersive Spectroscopy
- iii. Utilization of Jeffries method for quantitative image analysis

## **1.3 Project Background**

More and more cars are produced nowadays. It has become a major concern that the weight of cars has increased over 60% the past 26 years. Hence, this phenomenon has contributed to the global green house effect by the heavy emission of carbon dioxide, CO<sub>2</sub> to the atmosphere, as the fuel usage is correlative to the weight of vehicle.

The commercialization of aluminium usage has led to substantial increment by year, making it the world's second most used metal after steel [9]. Aluminium is the metal of choice for leading designers, architects and engineers, all of whom are looking for a material which combines functionality and cost-effectiveness with forward looking form and design potential.

Work-hardened wrought Al-Mg alloy 5xxx series has been commercially extended as the automotive Body in White (BIW) material (see Figure 1.1). Aluminium BIW is typically 40%-45% lighter than equivalent steel structure. A study done by Ford, BMW, Argonne, Ross, EAA and International Aluminum Institute [8] shown that, for every 10 % reduction in vehicle weight, there are 5% - 10% fuel savings. However, the reduction of mechanical strength (i.e. softening/thermal recovery) happens at elevated temperatures that allow easy dislocation repositions [9]. The softening behavior of Al-Mg alloy through aluminium alloy AA5083 series can be investigated through the observation and characterization of intermetallic particles ( $\alpha$  and  $\beta$  particles) in the aluminium matrix under different temperature of heat treatment.

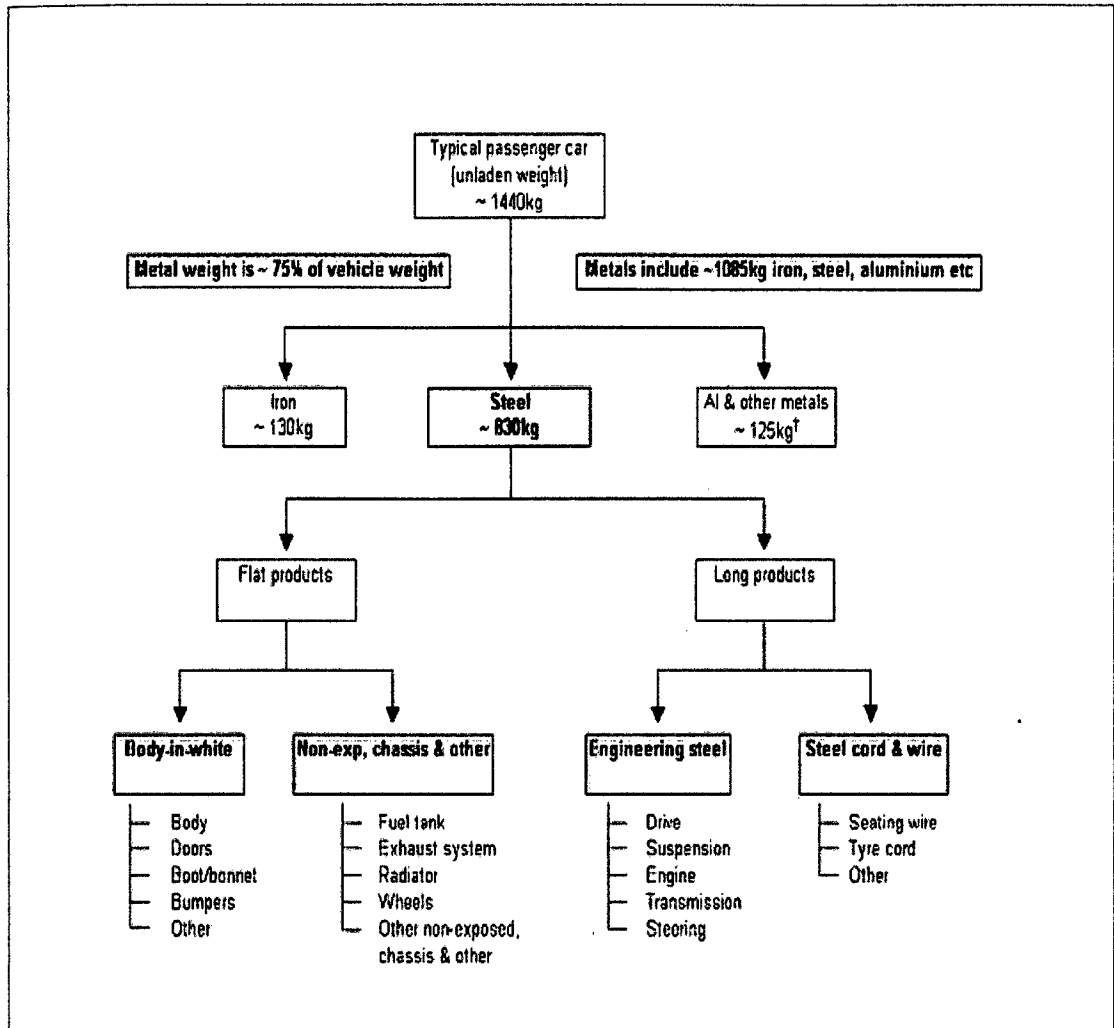


Figure 1.1 Comparison of different metal usage in car component [10]

#### 1.4 Project Gantt Chart

Refer to Appendix A

## CHAPTER 2

### LITERATURE STUDY

#### 2.1 Wrought Aluminium Alloy 5xxx series

Aluminium is the third most abundant metallic element on the earth [9] and always occurs in combination with other elements such as oxygen and silicon. However, aluminium is extremely useful in engineering field as it posses a combination of critical properties especially for transportation application. Although pure aluminium has low strength, it can be alloyed to strength up to 690Mpa.

The current aluminium based alloy that is used as body in white (BIW) for automotive is known as the 5xxx series work hardened wrought Aluminium Magnesium alloy. Aluminium Magnesium alloy that are used in BIW applications have very good formability; relatively low yield stress and work harden during cold working [11]. Magnesium is the principal alloying element and is added for solid-solution strengthening in amounts up to about five percent.

Alloy grades that have been used thoroughly as automotive BIW material are listed in Table 2.1. The alloys are nominally supplied to the BIW press shop in an annealed temper, designated as –O temper. The O temper is by means of annealed and recrystallized [3], which it is tempered with the lowest strength and highest ductility. The microstructure are characterized by a recrystallized grain structure as shown in Figure 2.1 and populated by iron (Fe) rich and magnesium (Mg) rich intermetallic particles and dispersoids [12] as shown in Figure 2.2.

The chemical element composition contained in commonly used 5xxx series aluminium alloys is shown in Table 2.1. Accordingly, the alloying elements (Refer Table 2.2) have significant influence on the mechanical properties of the 5xxx aluminium alloys [13]. Table 2.3 illustrates that the solubility of the main alloying element is relatively limited mainly if considering that iron and silicon are coming from the processing and are not (depending on the alloy) properly alloying elements. The classifying of the alloying elements in accordance to their solubility into three classes can be determined [2], which are:

- i. High solubility solutes (>10% at %): Zn: Ag: Mg: Li.
- ii. Intermediate solubility solutes (>1 and <10 at %) Ga: Ge: Cu: Si.
- iii. Low solubility solutes (<1 at %) all others

**Table 2.1** Composition of commonly used 5xxx series alloys used for automotive BIW components in weight percent with Al as remainder [13]

Alloy	Mg	Si	Cu	Fe	Mn	Zn	Cr	Ti
5251	1.7-2.4	0.40	0.15	0.50	0.10- 0.50	0.15	0.15	0.15
5052	2.2-2.8	0.25	0.10	0.40	0.10	0.10	0.15- 0.35	-
5754	2.6-3.6	0.40	0.10	0.40	0.50	0.20	0.30	0.15
5182	4.0-5.0	0.20	0.15	0.35	0.20- 0.50	0.25	0.10	0.10
5083	4.0-4.9	0.40	0.10	0.40	0.40- 1.00	0.25	0.05- 0.25	0.15

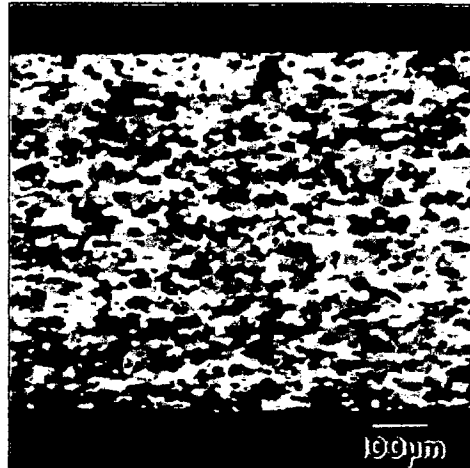
**Table 2.2** Mechanical properties of commonly used Al-Mg alloys used for automotive BIW components [13]

Alloy	Yield stress, 0.2% (MPa)	Ultimate Strength (MPa)	Tensile (UTS)	Maximum Elongation %
5251	80	180		25
5052	90	195		24
5754	100	215		24
5182	135	290		22
5083	145	300		22

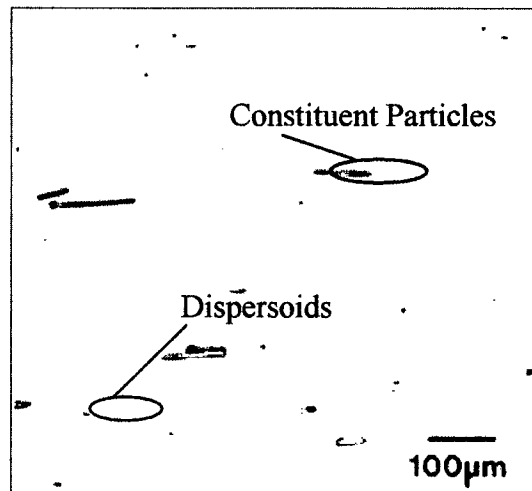
**Table 2.3** Solubility (wt %, temperature °C) of the main elements of Aluminium Alloys [2]

Alloying element	Maximum solid state solubility	Solubility at lower temperature	Atomic Radius difference (%)	Lattice Structure
Si	1.65 (577 <sup>0</sup> )	0.05 (250 <sup>0</sup> )	-6.3	Cubic; A4
Fe	0.052 (655 <sup>0</sup> )	0.001 (400 <sup>0</sup> )	-11.2	BCC/FCC
Cu	5.65 (548 <sup>0</sup> )	0.2 (200 <sup>0</sup> )	-11.2	FCC
Mn	1.82 (659 <sup>0</sup> )	0.36 (500 <sup>0</sup> )	-8.4	Cubic; A12
Mg	14.9 (451 <sup>0</sup> )	2.95 (150 <sup>0</sup> )	+11.9	CPH
Zn	82.8 (382 <sup>0</sup> )	4.4 (100 <sup>0</sup> )	-4.2	CPH





**Figure 2.1** Recrystallized grain structure of an annealed Al-Mg sheet [1]



**Figure 2.2** Constituent particles and dispersoids structure [11]

## **2.2 Wrought Aluminium Alloy AA5083 series characteristics**

In this study, wrought aluminium alloy AA5083 series is used as project specimen. Wrought aluminium alloys are non-heat-treatable aluminium alloys [13] as precipitation strengthening cannot be performed on them. This aluminium alloy can only be cold worked in order to increase their strength [3]. AA5083 alloy contains about 4.45 percent magnesium (Mg), 0.7 percent manganese (Mn) and 0.15 percent chromium (Cr) [14]. It is full-hardened temper, -H38 temper and is solution

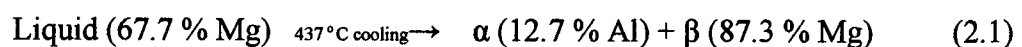
heat treated and naturally aged to a substantially stable condition-T4. H38 temper is by means of the alloy was strain hardened, then heated at low temperature to increase ductility and stabilized its mechanical properties. T4 is meant by the alloy was solution heat treated and naturally aged to a substantially stable condition.

### 2.3 Binary Aluminium Magnesium system

The Al-Mg binary alloy system consists of limited solid solubility in each other. The regions of restricted solid solubility at each end of the Al-Mg are designated as alpha,  $\alpha$  and beta,  $\beta$  phases. They are called terminal solid solutions since they appear at the ends of the diagram as shown in Figure 2.3. The alpha phase is an aluminium rich solid solution and can dissolve maximum of 14.9 wt % Mg at 451°C. The beta phase is magnesium rich solid solution and can dissolve maximum solid solubility of 87.3 wt% Al at 437°C (see Figure 2.4).

As the temperature lowered below 451°C, the maximum solid solubility of the solute elements decreased regarding to the solvus lines of Al-Mg phase diagram. The eutectic composition of the alloy composition freezes at a lower temperature known as eutectic temperature. It is where the liquid phase can exist in slow cooling. Thus, in Al-Mg system the eutectic composition (67.7% Mg and 32.3% Al) and the eutectic temperature (437°C) determine a point on the phase diagram called eutectic point.

So, when the liquid of eutectic composition is slowly cooled to the eutectic temperature, the single liquid phase transforms simultaneously into two solid forms,  $\alpha$  and  $\beta$  solid solution. In general, the transformation is known as the eutectic reaction as shown in Equation 2.1



During the progress of the eutectic reaction the liquid phase is in equilibrium with the two solid solutions  $\alpha$  and  $\beta$ , thus in this state three phases coexist and are in equilibrium. Magnesium can dissolve easily due to increasing temperature [2] rather than other alloying element in 5xxx series aluminium alloy, as seen in Table 2.2.

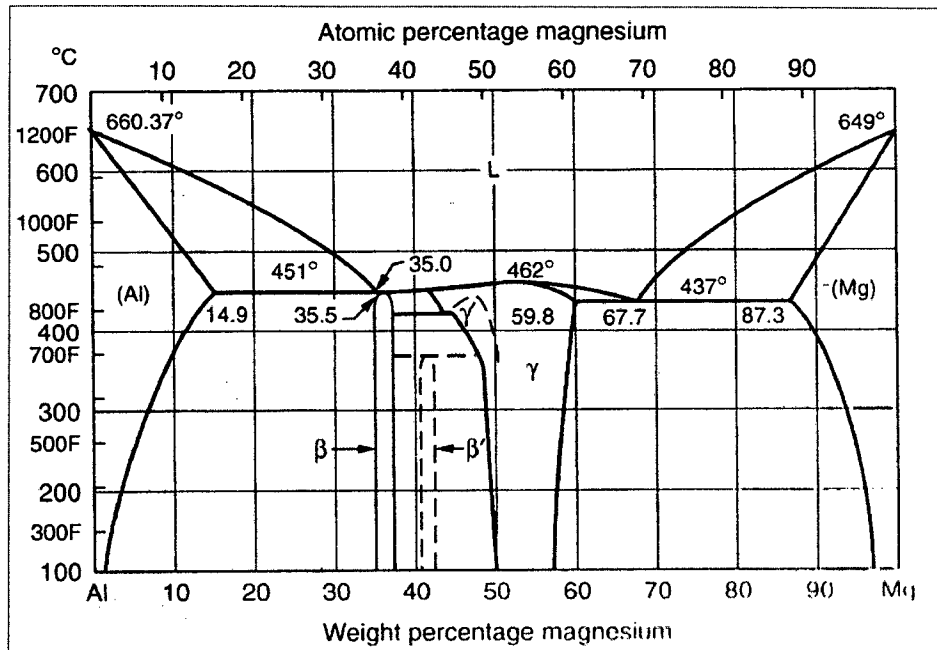


Figure 2.3 Al-Mg phase diagram [11]

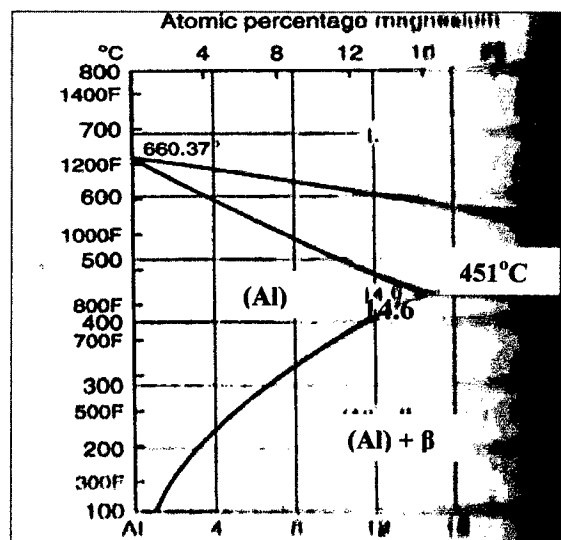


Figure 2.4 Aluminium rich end of Al-Mg phase diagram [11]

## 2.4 Intermetallic Particles

Intermetallic compounds (refer Appendix B) are the formation of two unlike metals diffuse into one another creating species materials (see Figure 2.5). Theoretically, intermetallic growth is the result of the diffusion of one material into another via crystal vacancies made available by defects, contamination, impurities, grain boundaries and mechanical stress [15]. It is a chemical compounds formed between metallic chemical elements that are nonmetallic in behavior. They are nonmetallic in behavior because they have ionic or covalent atomic bonding, which gives them low ductility and low electrical conductivity,  $Mg_2Sn$  for instance [13]. The intermetallic compounds should have a distinct chemical formula or stoichiometry (fixed ratio of involved atoms). Anyhow, in most cases a certain degree of atomic substitution takes place that accommodates large deviations from stoichiometry [2].

Intermetallic compounds which is also known as constituent particles, formed interdendritically by eutectic decomposition during ingot solidification. A group of insoluble compounds realized is that contains of impurity elements as iron and silicon [2, 13]. The examples are  $Al_6(Fe, Mn)$ ,  $Al_3Fe$ ,  $\alpha Al(Fe, Mn, Si)$ . The second group, which consists of equilibrium intermetallic compounds such as  $Mg_2Si$ ,  $Al_2Cu$  and  $Al_2CuMg$  is soluble constituents.

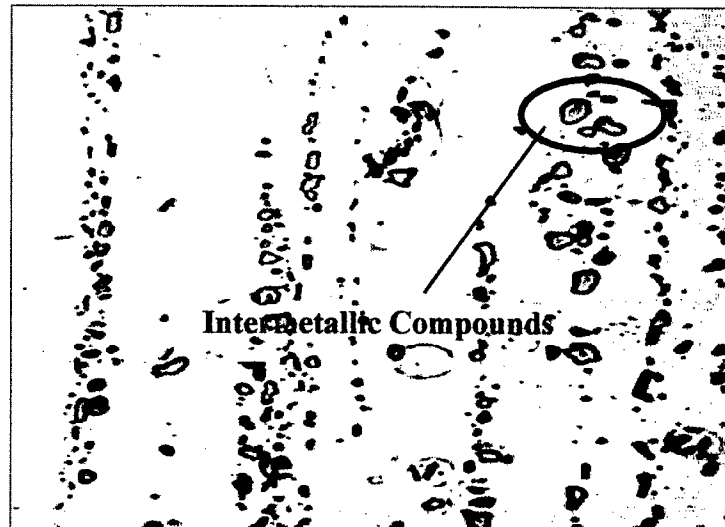


Figure 2.5 Intermetallic Compounds [13]

## 2.5 Dispersoids

Smaller submicron particles or dispersoids with typically  $0.05 - 0.5\mu\text{m}$  of size (refer Appendix B) that form during homogenization of the ingots by solid state precipitation of compounds consisting elements which have modest solubility and diffuse slowly in aluminium. These particles retard dissolution, coarsening, recrystallization and grain growth during processing and heat treatment of the alloy [14]. The compounds usually contain one of the transition metals such as  $\text{Al}_{20}\text{Mn}_3\text{Cu}_2$ ,  $\text{Al}_{12}\text{Mg}_2\text{Cr}$  and  $\text{Al}_3\text{Zr}$  [13].

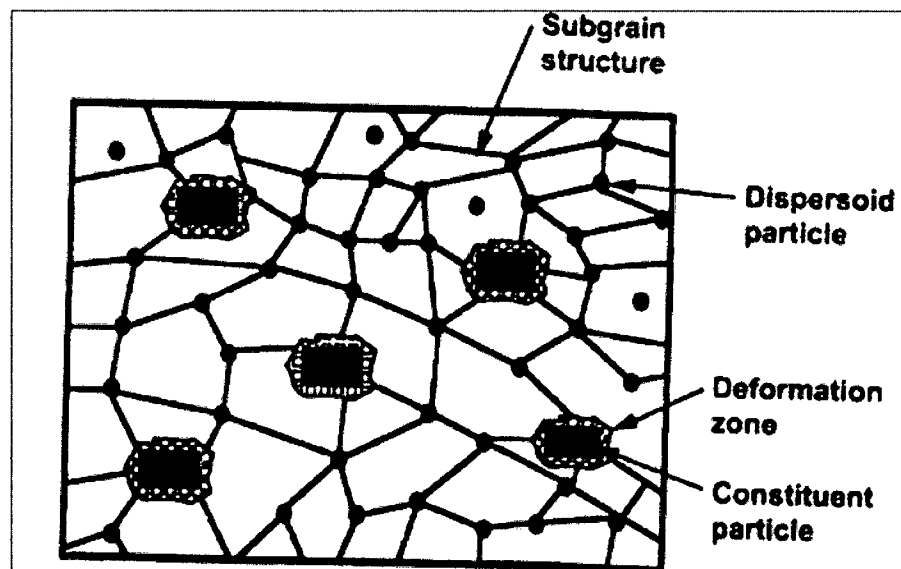
## 2.6 Strengthening mechanism

### 2.6.1 Work Hardening

There are 2% - 10% of energy employed in the deformation is stored in the metal in the form of lattice defects after cold working. The density and distributions of the defects, especially the dislocations, may be distributed non – homogeneously,

forming a cellular substructure. The stored energy and the dislocation density (and its distribution) are mainly affected by factors such as the metal in itself, its purity, grain size and deformation. The deformation bands formed as the high concentration heterogeneities in the distribution of lattice defects. It is also present as considerable variations in the orientation difference between regions about some micrometers apart. Basically, they are preferred locations for recrystallization start (nucleation).

Figure 2.6 schematically shows the dislocation substructure in an aluminium alloy containing primary particles of intermetallic phases and dispersoids. From the figure, the large strain concentration around the primary particles and the role of the dispersoids in blocking the cell walls that will transform into sub grain boundaries or sub-boundaries during subsequent annealing can be comprehended.



**Figure 2.6** Idealized cold worked substructure in an aluminium alloy containing both coarse and fine particles [11]

In a cold worked metal, the microstructural changes that occur are meant to reduce the stored energy due to straining. The energy decreased is provided by the

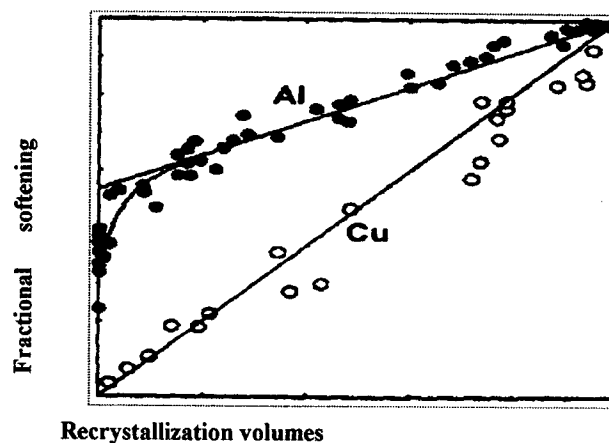
reduction and rearrangement of lattice defects. The major microstructural changes are as follows:

- i. Point defect reactions leading to the decrease of their number;
- ii. Annihilation of dislocations of opposite sign and shrinking of dislocation loops:
- iii. Dislocation rearrangement to attain smaller energy configurations (i. e. small angle boundaries):
- iv. Formation of high angle boundaries:
- v. Absorption of point defects and dislocations by migration of high angle boundaries:
- vi. Reduction of total grain boundary area.

The processes of (i) until (iv) are defined as recovery. The processes (v) and (vi) are defined as recrystallization and grain growth.

### **2.6.2 Recovery**

Before and even during the recrystallization of a cold-worked metal, the driving force for the migration of the high angle boundaries is decreasing continuously due to recovery. As the lattice defect distribution in the same sample is heterogeneous, a provided of more deformed micro region goes through the recrystallization process; some other neighboring micro region which is less deformed goes through the recovery process. Therefore, the regions that are not carried away by the migration of the high angle boundaries show a diminishing of dislocation density due to recovery (see Figure 2.7.).



**Figure 2.7** Fractional softening versus recrystallization volumes in copper (lower SFE) and aluminum (higher SFE) [11]

The lower the annealing temperature, the higher will be the participation of recovery in the global softening process. This is because of the recovery mechanisms caused smaller activation energies than those associated with the recrystallization mechanisms. If both processes are thermally activated and compete between themselves, lower temperatures favor the lower activation energy, i.e., recovery.

Recovery occurs through various mechanisms, starting at  $0.2 T_f$  ( $T_f$  is the absolute metal melting temperature). Recrystallization, on the other hand, occurs generally in the range of  $0.3$  to  $0.6 T_r$ . Therefore, when a metal is slowly heated up, the residence time at lower temperatures is greater, where exclusively recovery prevails. Consequently, the driving force for recrystallization will diminish due to the decrease in the quantity of lattice defects and to their rearrangement, thus delaying recrystallization

As the deformation temperature increases, recovery occurs with greater intensity, diminishing the driving force for recrystallization. In this study,