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Processing and precipitation strengthening of 6xxx series aluminium alloys: A review

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Abstract
This review paper gives a brief overview of processing stages and precipitation strengthening mechanism of 6xxx series aluminum alloys. 6xxx series aluminum alloys are widely used in defense and aerospace industries in different forms like sheet, rods, etc. These alloys have high strength in terms of strength to weight ratio. For industrial application these alloys, after direct chill (DC) casting, are homogenized and processed by different fabrication process like hot working, coldworking, process annealing, and strengthened by age hardening heat treatments. This results in the improvements of mechanical properties of 6xxx series alloys for industrial applications. These processing steps and the effect of the precipitations during age hardening are investigated through literature survey.

Keywords: 6xxx alloy, homogenized, processing, precipitation

Introduction

Aluminum is third most abundant elements or metals found in the earth crust as bauxite. It has a face centred cubic structure in metal form [1]. It is a silvery-white, soft, ductile and nonmagnetic metal. The usefulness of aluminum makes it the most broadly used metal after steel. Aluminum is one of the lightest engineering metals, having a strength to weight ratio higher to steel. It is broadly used for conductor cables and foils. Aluminum in its pure form is relatively soft and weak and thus it alloyed with a range of different elements to achieve required mechanical properties. The most important property of aluminum is its light weight having density 2.7 g/cm^3, which is about one-third of steel [2]. Aluminum alloy has excellent casting properties like low melting point, high degree of fluidity in molten stage and low shrinkage on solidification compared to steel. Aluminum alloys are replacing steels in applications where lower weight and reduced maintenance costs are requisites. Structural materials require not only high strength to weight ratio, besides reasonable cost, but also high fatigue resistance. Aluminum alloys have some other attributes like good electrical and thermal conductivities, corrosion resistance, good workability, ductility and strength, which make aluminum alloy widely usable and useful materials [1-2]. Because of its good formability and workability, it can be easily hammered and forged and can be drawn to any shape and size. Aluminum and its alloys when exposed to atmosphere, combines with oxygen to form a protective oxide coating which blocks further oxidation and protect the surface. The formation of protective oxide coating makes it highly corrosion resistant. If the protective layer of aluminum is scratched, it will instantly reseal itself [3].

In aluminum alloys, aluminum is the chief metal. To strengthen the pure aluminum other elements is added based on its application. In the resulting aluminum alloys, the typical alloying elements are copper, magnesium, manganese, silicon, tin and zinc. The properties of the aluminum alloys are modified by the addition of these alloying elements. Addition of various alloying elements effect microstructure and the mechanical properties of this alloys which is the principal method used to produce a selection of different materials that can be used in a wide variety of structural applications like in aerospace industries, transportation industries, satellite, etc. Aluminum alloys are classified as wrought and cast aluminum alloys which are further categorized into Non-Heat-Treatable and Heat-Treatable [2]. Non-Heat-Treatable (solid-solution-hardened) aluminum alloy did not respond to strengthening by heat treatment. 1xxx (pure aluminum), 3xxx (manganese), 4xxx (silicon) and 5xxx (magnesium) series of alloys falls under this category. Strength of these alloys are developed by stain-hardening or cold-working which is accomplished by cold rolling, drawing and other similar operations where area reduction is obtained during forming process.

Heat-Treatable (precipitate-strengthened) respond to strengthening by heat treatment. 2xxx (copper), 6xxx (silicon and magnesium) and 7xxx (zinc) series of alloys falls under this category. Heat treatment is employed to increase strength and hardness of the precipitation-hardenable wrought and cast alloys. Precipitation hardening alloys are temperature-dependent equilibrium solid solubility, characterized by increasing solubility with increasing temperature.

6xxx Processing

Processing of 6xxx series aluminium alloys plays an important role in determining its mechanical properties. Properties of aluminium alloys also depend on its microstructure, average grain size and its distribution, volume fraction of precipitate and the crystallographic orientation [4, 5]. Hence, processing and compositions are two important tools in determining the mechanical properties of this alloy. The heat treatable 6xxx series aluminium alloys are processes in the following steps: Casting and homogenization, hot working, cold working, annealing and ageing treatment.

Casting is one of the main routes for producing aluminium alloy ingot before processing. It is a process of melting the aluminium and its alloys in required quantity and pouring into a mould or die [6]. Before casting, fluxing or degassing of the melt is carried out to remove slag, oxides, gases and non-metallic impurities for obtaining a high-quality casting product. Homogenization treatment is the first basic processing step after casting of 6xxx series alloys and it is performed to the casting product/ingot at an elevated temperature, below the melting point of the aluminium alloy. Homogenisation treatment produces a homogenised structure by uniform distribution of the alloying elements and other constituents in the cast alloy. The homogenization treatment of 6xxx series aluminium alloys is in the range of 530 °C - 600 °C [7].

After casting and homogenization, hot working process is carried out which converts cast ingots into wrought products [8] like sheet, plate, rod, tubes, etc. Hot working process like rolling and extrusion is a plastically deformation process of heated alloy materials (billet) by application of a compressive force. The hot working temperature of 6xxx series aluminium alloy is around 468 °C [9]. Hot working results in poor surface finish. So, in order to improve the surface finish condition and strength, wrought aluminium alloys are cold worked at room temperatures after hot working process [8].

To eliminate the strengthening effect of cold working and to soften the aluminium alloy, annealing is done by heating the aluminium alloy at a temperature range from 260 °C to 440 °C. The time required to soften can vary from hours at low temperatures to seconds at high temperatures. The maximum annealing temperature of aluminium alloys is moderately critical and it is advisable not to exceed 415 °C, because of oxidation and grain growth [10].

In order to strengthen the 6xxx series aluminium alloy, the age hardening treatment is done. This treatment involves solutionizing at elevated temperature and then quenching to room temperature followed by room temperature aging or elevated temperature aging (artificial aging) in the temperature range. For 6xxx series aluminium alloy, the solution temperature is around 520 °C - 570 °C and artificial ageing temperature is around 160 °C - 200 °C [10].

6xxx Alloys System

In 6xxx series Al-Mg-Si alloys system, Mg and Si are the main alloying elements, thus making them heat treatable. This heat-treatable alloy may in the T4 temper condition (solution heat treated condition) and T6 temper condition (precipitation heat treatment). This group of alloys contain up to 1.5 % each of Mg and Si in the approximate ratio to form Mg_2Si, that is, 1.73:1 [2]. The maximum solubility of Mg_2Si in aluminium is 1.85%, and this decreases with temperature. A phase diagram of Al-Mg-Si alloys showing the solubility of Mg and Si as a function of temperature represented by the Mg_2Si phase are shown in figure 1. The ratio of the solubility of Mg and Si is crucial in 6xxx series and is responsible for the mechanical properties of the alloy. The well optimum behaviour of alloys can be obtained at a Mg/Si solubility ratio close to 1.74.

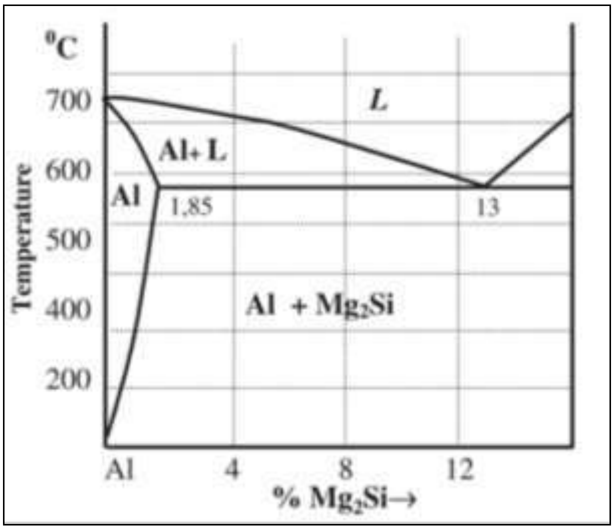

Fig 1: Aluminium- Magnesium Silicide Phase Diagram [14]

6xxx series aluminium alloys can be divided into three groups depending on the total amount of magnesium and silicon content. The alloy that fall under the first group, its total amount of magnesium and silicon does not exceed 1.5% in weight. 6063 is the aluminium alloy under this group with 1.1% Mg_2Si. In the second group the total Mg and Si contains exceed 1.5%. It contains Cu about 0.3% that enhance its strength in the T6 temper. Other elements such as chromium, manganese and zirconium are used for controlling grain structure. 6061 is the aluminium alloy that fall under the second group. The third group like 6009, 6010, and 6351 contain excess of silicon. An excess of 0.2% Si increases the strength of an alloy containing 0.8% Mg_2Si [2]. The composition of some 6xxx series alloys [20] are shown in table 1.

Characteristic of 6xxx series alloy is complex because of different sets of phases present in different areas of an ingot due to the gradient of cooling rate [11]. In equilibrium, aluminium solid solution consists of Al_8Mg_5, Si and Mg_2Si phases [1, 13]. The Mg_2Si phase (63.2% Mg, 36.8 %Si) has a cubic structure with lattice parameter, a = 0.635 - 0.640 nm. Low solubility Mg_2Si phase has a density of 1.88 g/cm^3 and a melting point of 1087 °C [1]. Even if the mutual solubility of Mg and Si in solid aluminium is low, it enables a significant effect of precipitation hardening. This is due to formation of metastable coherent and semi coherent β″ and β′ precipitates of Mg_2Si phase during ageing [15]. It is important to mention that the composition of these metastable coherent precipitates is different from that of the equilibrium Mg_2Si phase. β″ precipitates has an excess of silicon and it was identified as Mg_5Si_6 [16].

Table 1: Composition of some 6xxx series alloys in wt.% [20]

6xxx series	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	others	Balance Al
6003	0.35-1.0	0.6	0.10	0.8	0.8-1.5	0.35	0.20	0.10	0.15	Rem.
6005	0.6-0.9	0.35	0.10	0.10	0.40-0.6	0.10	0.10	0.10	0.15	
6010	0.8-1.2	0.50	0.15-0.6	0.20-0.8	0.6-1.0	0.1	0.25	0.10	0.15	
6013	0.6-1.0	0.50	0.6-1.1	0.20-0.8	0.8-1.2	0.1	0.25	0.10	0.15	
6055	0.6-1.2	0.30	0.50-1.0	0.10	0.7-1.1	0.20-0.30	0.55-0.9	0.10	0.15	
6061	0.40-0.8	0.70	0.15-0.40	0.15	0.8-1.2	0.04-0.35	0.25	0.15	0.15	
6063	0.20-0.6	0.35	0.10	0.10	0.45-0.9	0.10	0.10	0.10	0.15	
6081	0.7-1.1	0.10	0.50	0.10-0.45	0.6-1.0	0.10	0.20	.015	0.15	
6082	0.7-1.3	0.50	0.10	0.40-1.0	0.6-1.2	0.25	0.20	0.10	0.15	

Iron presents in Al-Mg-Si as impurities and it is difficult to eliminate. Fe along with Mg and Si in this alloy system, can produces a quaternary compound. This heat resistant quaternary compound was designated as π with formulae $Al_8FeMg_3Si_6$ (10.9 % Fe, 14.1% Mg, 32.9% Si) and density 2.82 g/cm^3 [1, 17]. It has hexagonal crystal structure with lattice parameters, a = 0.663 nm and c = 0.794 nm. Depending on the concentration, other binary and ternary systems like Al_3Fe, Al_8Mg_5, Mg_2Si, Al_8Fe_2Si, Al_5FeSi can be in equilibrium with the aluminium solid solution [1-13]. The Mg_2Si is in equilibrium with all other phase and occurs in most alloys in the solid state. Manganese addition to 6xxx alloys mitigate the harmful effects of acicular β-AlFeSi by transforming it to the more favourable globular α-Al(Fe,Mn)Si [18]. Iron correction by Mn can increase toughness. In addition to Mn, 6xxx may also have other trace elements such as Ti, Cr, Zn, Cu and some of which form dispersoids during homogenization and post homogenization quenching [19]. Mn and Cr increase the strength while titanium contribute to hardness of the alloy. Zirconium, usually less than 0.15% is added to form Al_3Zr dispersoids for recrystallization control [2].

Homogenisation of 6xxx Aluminium Alloy:

Homogenization of the aluminium alloys is the process of heating to a suitable temperature (530 °C to 600 °C) [7] after casting which followed by soaking and cooling. Homogenisation is the first step after casting before doing any hot working operations like rolling and extrusion. During solidification of the as cast Al-Mg-Si alloy, intermetallic phases which mainly consist of β-AlFeSi and coarse Mg_2Si occurs. These intermetallic phases occur in the dendritic microstructure of the as cast Al-Mg-Si alloy which generally consist of Fe due to the low solubility of iron in aluminium [9]. The plate shape β-AlFeSi particles reduces the ductility of the alloy. Due to these non-homogenised intermetallic phases, the workability of the as cast alloy reduced [21, 22]. The βAl5FeSi phase can be detrimental for extruded products. Therefore, there is a need to have a high homogeneity with uniform structure and minimum amounts of platelets like β-AlFeSi and coarse Mg_2Si particles in the as cast billet [23]. Though it is difficult to avoid the formation of these phases during solidification but by homogenisation their negative effect can be reduced. During homogenisation morphology of platelets like β-AlFeSi phase transformed to Chinese script α-AlFeSi phase along with dissolution of Mg_2Si particles [24, 25]. The round shaped α-AlFeSi particles formed during homogenisation provide better condition to workability [26, 27]. The purposes of homogenization can be summarized as follows:

- To homogenize the concentration of β-AlFeSi and Mg_2Si particles which weakens the structure by locally melting in the grain boundary during extrusion or rolling,
- Dissolution of low melting-point Mg_2Si and Si particles formed during solidification, which cause tearing during extrusion,
- To transform β-AlFeSi to thermally stable α-AlFeSi particles, which has less change of local melting during extrusion,
- Spheroidization and Fragmentation of α- AlFeSi particles, which will result in a better extrusion property [27, 29].

Therefore, the homogenization, a heat treatment process, is usually used to improve billet quality by fragmenting and transforming the needle shape β-Al_5FeSi to a more spheroidal α-Al_8Fe_2Si. Whereas, the Mg2Si particles dissolved in an aluminium matrix during homogenization [30, 32]. The Fe:Si ratio in the range of 1.28-3.83 is used to predict α-Al_8Fe_2Si particle [33] while the minimum Fe: Si atomic ratio of 1.00 or 1.00 ± 0.25 can be used to predict β-Al5FeSi particle [33, 34]. In addition, for a better prediction of β to α transformation during homogenization of Mn-containing 6xxx alloys, the Fe(+Mn): Si should be used for calculation [22, 35]. Fig. 2 shows the optical micrograph of dendritic microstructure of as cast 6063 aluminium alloy showing intermetallic phases at the grain boundary. Details of β-AlFeSi and Chinese script α-AlFeSi intermetallic phases can be seen in as cast condition.

The homogenization temperature is a critical factor in determining extrudability and surface appearance of the extrudate by converting β-AlFeSi to thermally stable less detrimental α-AlFeSi phase. The β to α transformation involves loss of Si from β phase to the matrix which makes the α-iron phase less brittle and also provides a greater amount of Mg_2Si for subsequent precipitation [36]. The time required for homogenization at a defined temperature is largely depended on the size and amount of the AlFeSi phases in the as-cast microstructure. Micro-segregation or non-uniformity in composition of Si and Mg is observed to be greatly reduced at temperatures in excess of 550 °C because of high diffusivities of Si and Mg in aluminium. Thus, a better mechanical properties and good surface finish were observed by homogenizing at 560 °C for 6 hours in extruded in 6063 aluminium alloy. At this homogenization time and temperature, also a high degree of spheroidization of the α-AlFeSi compound observed in 6063 aluminium alloy [9]. Raising the temperature to 580 °C, the β-AlFeSi phase fully transform to the α-AlFeSi phase in the 6063-aluminium alloy with a very low Mn level (0.004 wt.%) [37].

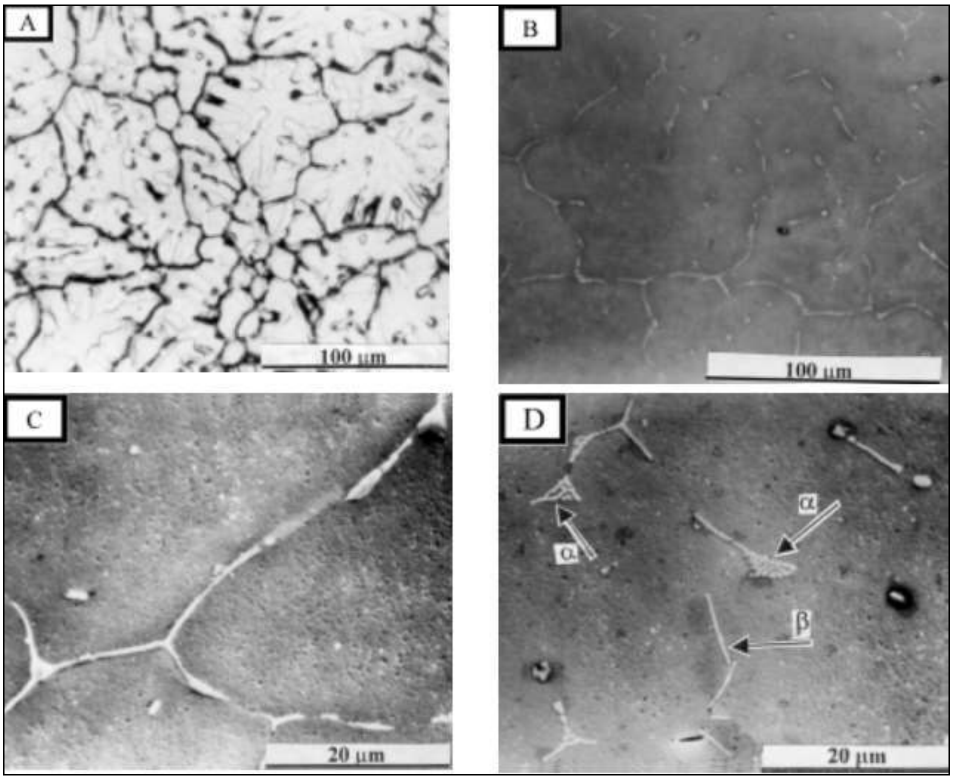

Fig 2: Microstructure of AA-6063 A) optical micrograph of cellular dendritic solidification B) SEM image of intermetallic phases at grain boundaries C) and D) Detail microstructure showing morphology [9].

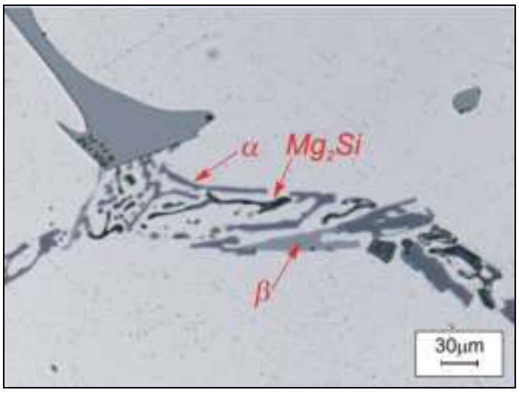

Fig 3: Optical Micrograph of as cast 6082 billet showing complex intermetallic particles [38].

Fig. 3 shows a higher magnification optical micrograph of as cast microstructure of 6082 billet. Mg_2Si particles appears dark and plate like β-AlFeSi particles and needle or rode type α-AlFeSi particles appears light [38]. Fig. 4 shows the morphology of a cast plate like β-AlFeSi and Chinese script α-AlFeSi phases. Fig. 5(a) shows as-cast 6063 ingot showing iron-rich phases (light) and Mg_2Si (dark) in dendrite interstices. During homogenisation of the ingot Mg_2Si has been solutionised, leaving slight spheroidized iron-rich phases, Fig. 5 (b). During homogenizing at 585 °C for 60 min the average grain size increases to 104µm and 108µm. Average grain size of 6063 aluminium alloy varies with varying Fe content. The average grain size was found to be 74µm and 93µm for 6063 cast alloy containing 0.089 wt. % and 0.17 wt.% Fe [39].

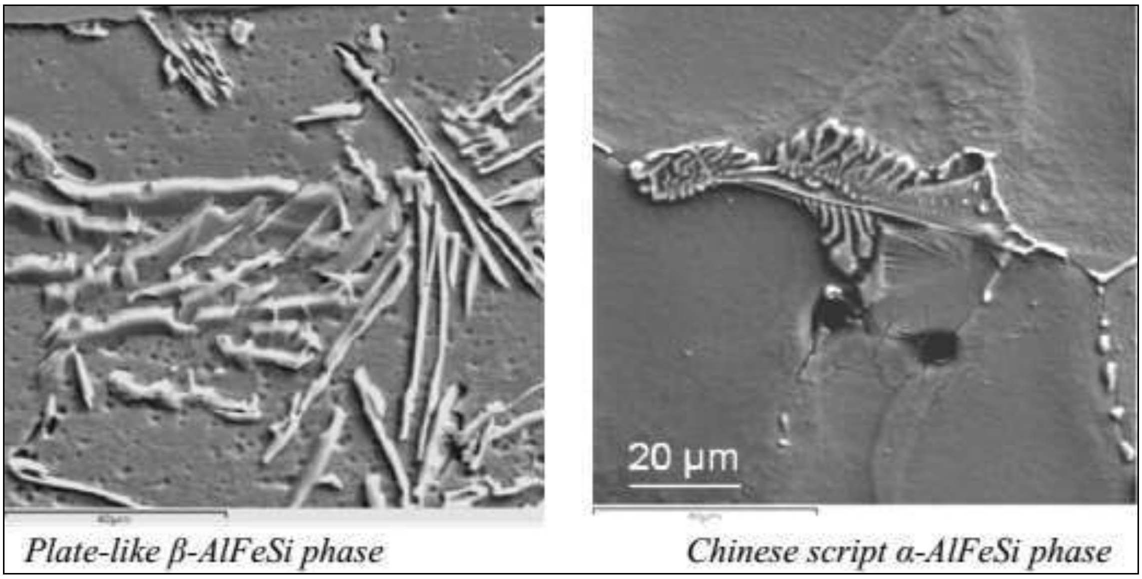

Fig 4: SEM micrograph showing different morphology of as cast β and α phases AlFeSi in 6063 aluminium alloy [24].

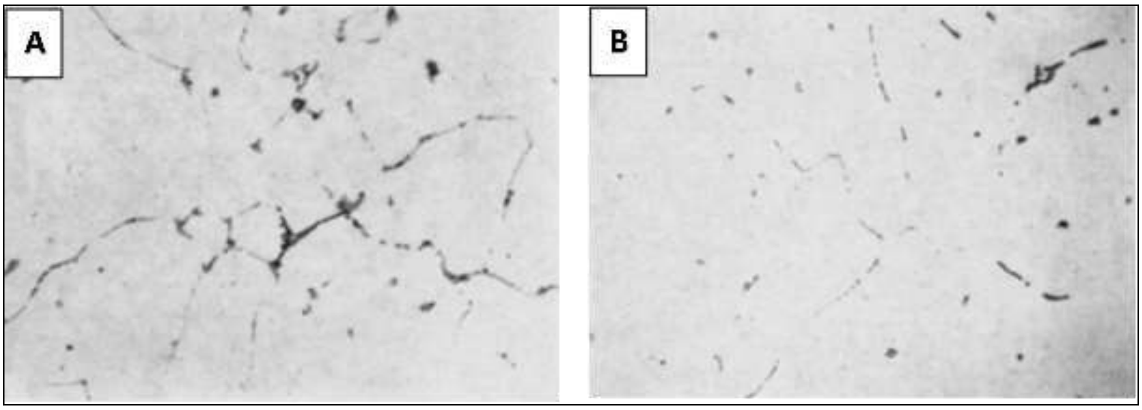

Fig 5: Microstructure of AA-6063 (A) as-cast ingot (B) Homogenised ingot, 0.5% hydrofluoric acid, 445 X [25].

Precipitation Strengthening of 6xxx Alloys

The strength of an alloy depends on the ability of precipitates and atoms in solid solution, to obstruct the motion of dislocations. Duralumin is the first precipitation/age-hardened aluminium alloy which was patented by Alfred Wilm in 1909. The strength of duralumin increased by more than doubled due to precipitation strengthening [40, 41]. The strengthening of Al-Mg-Si alloys is carried out in three steps viz., solution heat treatment, quenching and precipitation hardening or artificial ageing.

Solution heat treatment was carried out to dissolved the solute and produce a nearly homogeneous solid solution. This result in a single-phase region. The temperature of solution treatment depends on the composition and maximum solid solubility of Si in the alloy [10] and it between the melting point and the solvus temperature of the alloy.

Then after sufficient time of STT, the alloy is quenched to room temperature. Rapid cooling prevents the alloying elements from precipitating, also results in formation of vacancies and solutes in the Al matrix as much as possible. The alloy is now attaining the supersaturated solid solution (SSSS) state. So, we have a super saturated solid solution of magnesium, silicon, and other elements in aluminium at room temperature. This is called the T4 temper. This state is highly unstable even at room Temperature.

For precipitation hardening, vacancies are required to form the precipitates. Thus, the super saturated solid solution (SSSS) results to the formation of these vacancies. Presence of vacancy increases diffusion and results in the formation of solute clusters [42, 43] and dislocation loops [44-46]. Due to solute clustering and coarsening, strength of the alloy was reported to decrease [47]. During ageing [42] Si forms initial clusters, attracting slower Mg atoms, causing co-clusters to form, which then further grow into Guinier - Preston (GP) zones. GP zones is the ordered arrays of atoms in the aluminium matrix that strengthen the aluminium considerably. GP zones (also called clusters by some authors) are believed to be spherical with uncertain structure. The precipitation sequence in ternary, unstrained Al-Mg-Si alloys is generally given as [48, 49].

α (SSSS) → atomic clusters → Guinier-Preston zones (pre β″) →β″ → β′/ U1/ U2 /B → β, Si (stable).

The crystal structure of the most common precipitate phases in Al-Mg-Si alloys are shown in Table 2 with corresponding compositions, morphology and crystal structure.

Table 2: Overview of precipitate phases in the Al-Mg-Si system

Precipitation Sequences	*Composition*	*Morphology*	*Crystal structure*	*Ref.*
SSSS	Unknown	Point Defect	FCC	49
Atomic Cluster	varying Mg and Si contents	Cluster	Unknown	49
GP zone	$Mg_xAl_{5-x}Si_6$	Spherical	Primarily monoclinic	16
β″	Mg_5Si_6	Needles	Body cantered monoclinic	50
β′	$Mg_{1.8}Si$	Rods	Hexagonal	51
U1	$MgAl_2Si_2$	Needle	trigonal	52
U2	MgAlSi	Rods	orthorhombic	53
B′	$Mg_{48}Al_6Si_{36}$	laths	hexagonal	54
β	Mg_2Si	Plates or cubes	FCC	55

During heat treatment solute atoms are distributed in a supersaturated solid solution (SSSS). SSSS start decomposing as Mg and Si atoms attract each other which is referred as atomic clustering. This phenomenon happens during natural ageing or room temperature storage. When artificial ageing i.e. heating at a higher temperature above room temperature, fully coherent Guinier Preston (GP)-zones which are believed to be spherical with uncertain structure start to develop. GP-zones are sometimes also called initial-β″. During artificial ageing when the hardness of the material reaches maximum, high density of finely dispersed GP-zones and β″ type precipitates are found to be dominant in the microstructure of the Al-Mg-Si alloy. The β″ term represent fine needle-shaped precipitates which contributes most to mechanical properties when densely dispersed. The structure was determined as monoclinic type of Mg_5Si_6. This precipitate growing along a <100> direction of the Al matrix [56]. Further, ageing beyond peak hardness i.e. over ageing, results to transformation of β″ to a number of other metastable phases like β′, B′, U1, U2. The β′ rod like precipitates that grows from the β″ category have a negligible contribution to mechanical properties. These are thermally more stable and coarser than β″ with a length of ~500 nm [48]. Hexagonal structure β′ also grows along one of the <100> directions of the Al matrix [57,58]. Later, stable equilibrium phases β and Si are obtained after sufficient ageing time. The β phase are coarse Mg_2Si in platelets form or cube-like in shape that grows from the β′ phases. These phases due to its large size, has no contributes to mechanical properties of the alloy. Fig. 6 shows the strength evolution with ageing time of 6xxx-alloys [8].

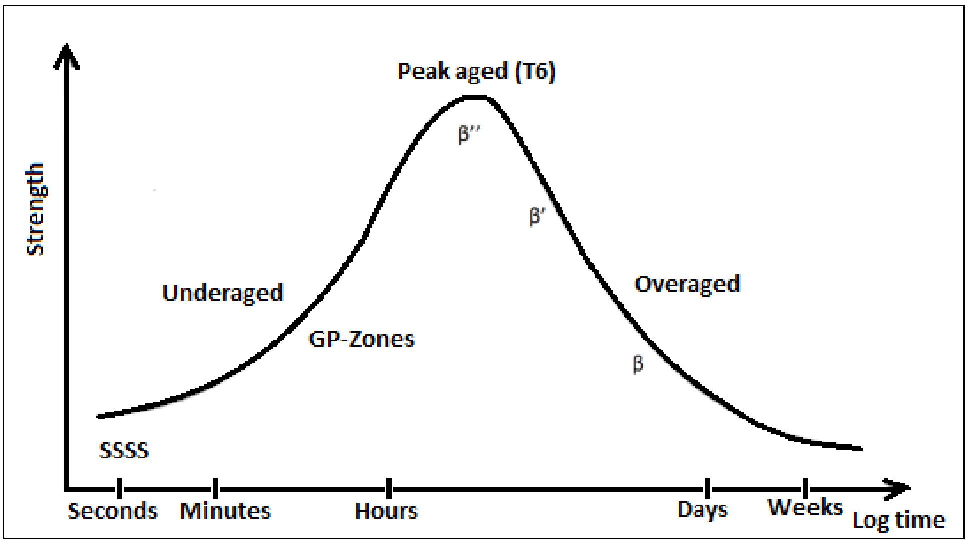

Fig 6: Strength evolution during artificial ageing of an AlMgSi alloy [59].

TEM examinations on 6063 aluminium alloy at underaged (373 K, 8 h), peak-aged (448 K, 8 h) and overaged (523 K, 8 h) was carried out by Shekhar et.al [59]. Underaged conditions reveals the likely clustering of solute atoms and some dislocations as shown by Fig. 7(a). Peak aged alloy in .7 nm respectively.

Fig. 7(b) show the formation of needle shape β″ type precipitates having average dimensions as 62.8 nm × 5.8 nm and overaged alloy in Fig. 7(c) shows the formation of needle shape β type precipitates having average dimensions 500 nm × 35

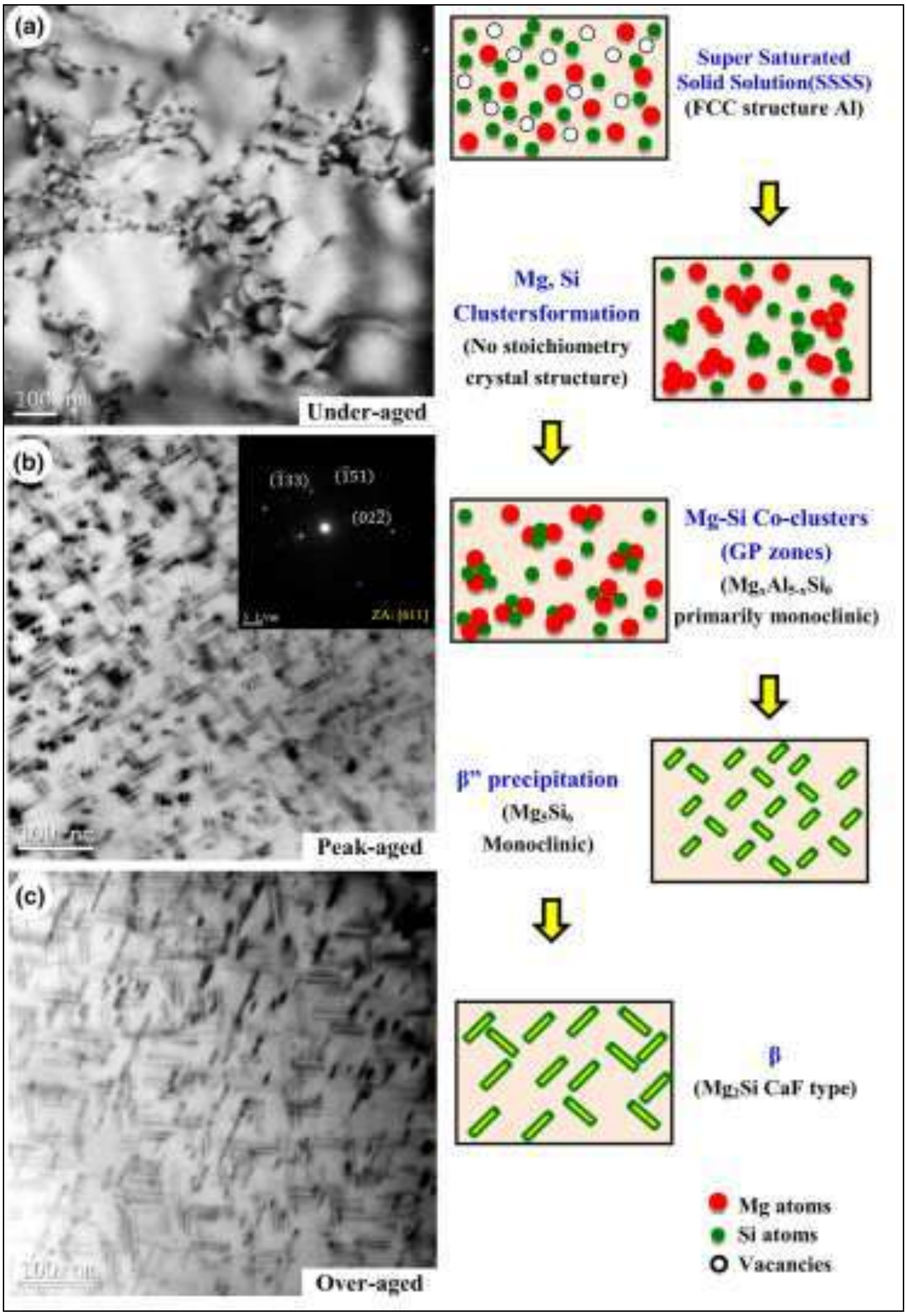

Fig 7: Representative bright field TEM micrographs of 6063 alloy corresponds to (a) underaged (373 K, 8 h), (b) peak-aged (448 K, 8 h) and (c) overaged (523 K, 8 h) states. Sequence of aging process is schematically illustrated in the right column [59].

Conclusions

This review shows that for enhancing the mechanical properties of 6xxx series aluminum alloy proper it is important to understand and follow the proper processing steps and also its mechanism behind it strengthening. It is well-known from this review that proper homogenisation is the basic important steps before processing of 6xxx series aluminium alloys. Homogenisation improve cast billet quality by breaking down and transforming the needle shape β-Al_5FeSi to a more spheroidal α-Al_8Fe_2Si and dissolving the hard Mg_2Si particles in an aluminum matrix. Later, the alloy is processed and strengthened by precipitation of high density of finely dispersed GP-zones and β″ type precipitates.

Conflicts of interests: All authors declare no conflicts of interest in this paper.

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