CHAPTER 5 MICROSTRUCTURES AND STRENGTH OF POWDER-THIXOCAST AL-25SI-2.5CU-1MG AND AL-20SI-5FE ALLOYS

5.1 Motivation

Chapter 4 has illustrated the feasibility of the novel process, powder thixocasting, in net-shape forming of a hypereutectic Al-Si-Cu-Mg alloy with fine and uniform primary Si particles in. However, material properties of those alloys fabricated by powder thixocasting are still needed to be investigated and compared with conventional alloys.

In this Chapter, the powder-thixocast alloys include Al-25Si-2.5Cu-1Mg and Al-20Si-5Fe; whereas, the conventional alloys used for comparison include an Al-25Si-2.5Cu-1Mg alloy fabricated with traditional ingot thixocasting route and an Al-12Si-1Cu-1Mg (LM13) alloy fabricated with liquidus casting routes.

Experimental results show that powder-thixocast Al-25Si-2.5Cu-1Mg alloy has much finer microstructure and slightly higher strength values than the conventional alloys. However, powder-thixocast Al-20Si-5Fe alloy has lower strength than the conventional alloys, due to its little precipitation hardening effect; despite it also shows preferable fine and uniform distributed intermetallic compound particles.

5.2 Experimental

5.2.1 Materials preparation

Table 5.1 lists four hypereutectic Al-Si-X alloys that were used herein, two were fabricated by powder thixocasting (PT), and two were by conventional routes. Al-25SiCuMg (IT), having similar compositions to Al-25SiCuMg (PT), was fabricated using a conventional process termed as “ingot thixocasting” herein. LM13, the most popular alloy for automobile pistons up-to-date, was fabricated by pouring a superheating melt into a permanent metallic mould and then being solidified under atmosphere pressure. Finally, fabrication of Al-25SiCuMg (PT) has been detailed in

Chapter 4.

Table 5.1 Chemical compositions of the Al-Si-X alloys

Alloy	Processing route	Chemical composition (wt%)							
		Si	Cu	Mg	Mn	Fe	Ni	Ti	Al
Al-25SiCuMg (PT)	Powder thixocast	24.6	2.56	1.04	0.47	0.16	0.01	0.03	Bal.
Al-20SiFe (PT)	Powder thixocast	20.0	0.02	0.02	.01	5.64	—	—	Bal
Al-25SiCuMg (IT)	Ingot thixocast	24.2	2.25	1.08	0.08	0.69	0.04	0.08	Bal
LM13	Gravity cast	11.6	1.08	1.12	0.01	0.50	1.00	0.03	Bal

The following sections will detail the fabrications of Al-20Si-5Fe (PT) and Al-25SiCuMg (IT) alloys.

A. Fabrication of Al-20Si-5Fe (PT) by powder thixocasting

The hypereutectic Al-20Si-5Fe powder, supplied from Valimet Inc, USA, was fabricated by a gas-atomization process. Its particle size values of d_{10}, d_{50}, d_{90}, measured using a particle size analyzer (Coulterr), are 150, 193 and 270μm, respectively.

The Al-20Si-5Fe powder preforms were consolidated at about 575 ^{o}C Then, the preforms were heated to a semisolid temperature by an induction coil. Afterward, they were immediately transferred to a sleeve and extruded into a mould cavity by a plunger of a die-casting machine. All the above equipments used are described in Chapter 4.

In fact, Al-20Si-5Fe alloy was found less suitable for thixocasting than Al-25Si-2.5Cu-1Mg alloy. Firstly, it is because that the former alloy has no stabilizer of α−Al grains, such as copper in the latter alloy. When the latter alloy's temperature is controlled within 523 ^{o}C and 565 ^{o}C, the ternary phases of α−Al grains, primary Si particles and melt coexists. Thus, liquid fraction in this alloy is still small but is sufficient for thixocasting, as has been detail in Chapter 4.

The second reason is that the suitable temperature range for thixocasting Al-20Si-5Fe alloy is very narrow, just from about 576 ^{o}C to 577 ^{o}C. Figure 5.1 shows a typical DTA scan of Al-20Si-5Fe powder. Upon heating, the scan only has one onset point at 576 ^{o}C, which is different from that scan of Al-25Si-2.5Cu-1Mg has two onset point at 523 ^{o}C and 565 ^{o}C (Fig. 4.16).

Table 5.2 shows the possible reactions during solidification of Al-20Si-5Fe alloy [102-109]. It can be found that addition of Fe in Al-Si alloys results in a ternary reactions 5 and 6 in Table 5.2. However, their reaction temperatures are 575 ^{o}C and 573 ^{o}C, which are too close to the eutectic point at 577 ^{o}C of the eutect reaction, Liquid →Al + Si. Therefore, Al-20Si-5Fe is only suitable to be thixocast at temperature within 573 ^{o}C to 577 ^{o}C, at which α−Al grains can coexist with primary Si particles and a small amount of liquid. However, this temperature range is too narrow; besides the amount of liquid at the temperature range should be very small since the amount of the ternary reactions 5 and 6 in Table 5.2 should be very small, because that no additional peak was found between 573-577 ^{o}C in Fig. 5.1.

Actually, in our experience, Al-20Si-5Fe alloy powder preforms were necessarily heated higher than 580 ^{o}C for semi-solid forming. At such high temperatures, all the α−Al grains will melt and only the primary Si particles remain in solid in the alloys, indicating liquid fraction was high to about 80% during thixocasting. Such a high liquid fraction, however, caused that the powder-thixocast Al-20Si-5Fe samples had coarse primary Si particles due to slow solidification, and defect of porosities due to turbulent flow or solidification shrinkage. These porosities, however, did not present in Al-25SiCuMg alloy, indicating suitable constitutions is necessary for powder thixocasting.

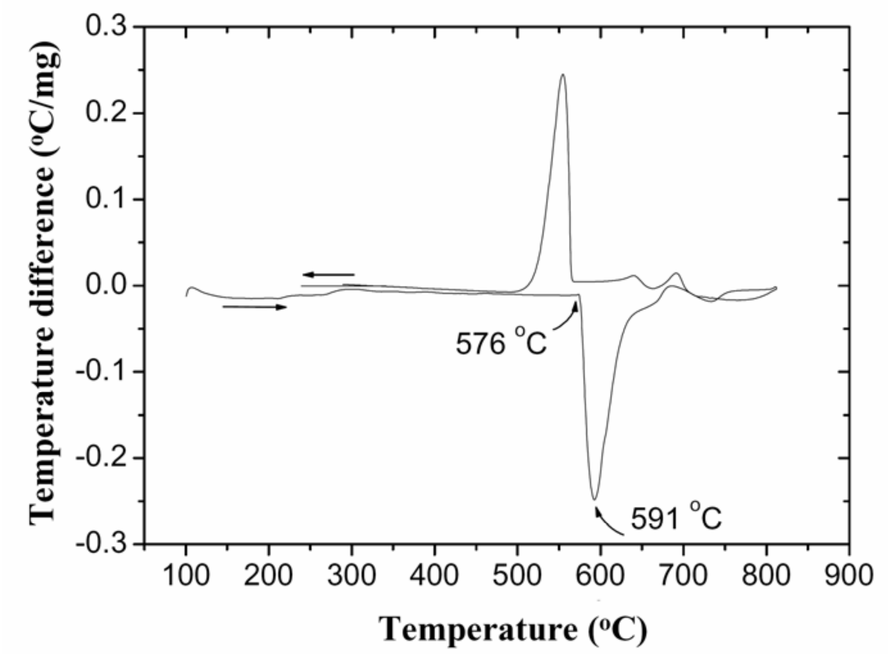


Figure 5.1 A typical DTA scan spectrum of Al-20Si-5Fe powder.

Table 5.2 Possible reactions during solidification of Al-20Si-5Fe alloy

No.	Reactions	Composition		Temperature
		%Si	%Fe	(°C)
1.	Liq.→Liq. + δ-Al_4FeSi_2 [103]			~760
1	Liq. →Liq. +Primary Si* [102]	20	--	700
2	Liq.→Liq. + $FeAl_3$*[102]	--	5	680
3	Liq.→Al + $FeAl_3$[104]	--	1.8	655
4	Liq.→Al + Si [102]	12.6	--	577
5	Liq.→Al + Si + Al_5FeSi [105]			575
6	Liq.→Al + Si + $Al1_5(Mn,Fe)_3Si_2$ [105]			573

* Estimated from binary Al-Si or Al-Fe phase diagram [102].

B. Fabrication of Al-25SiCuMg (IT) by ingot thixocasting

The process name of “ingot thixocasting” used here is to contrast with that of “powder thixocasting”. In the former process, the feedstock billets used for thixocasting were fabricated by ingot metallurgy (IM), while the latter were by powder metallurgy (PM). The abbreviation of IT refers to ingot thixocasting, and PT refers to powder thixocasting.

Traditionally, the thixocasting Al-Si alloy billets are fabricated by melt-stirring method, as is described in Chapter 2. However, in this study, the billets used for Al-25SiCuMg (IT) alloy was prepared by squeeze casting.

A melt of the hypereutectic Al-Si-Cu-Mg alloy without any refiner for primary Si was poured at 800°C into a steel mould and pressed using a plunger at a pressure of 85 MPa until the melt solidified. The cast billets have a diameter of 76 mm and a length of 85 mm. Afterward the cast billets underwent a similar thixocasting process and used a similar mould that was adopted for the powder preforms.

Method of fabricating SSMF feedstock by squeeze casting is similar to that of "rapid slug cooling technology" (RSCT) proposed by M. Fehlbier and co-worker [28]. They found that a "thixotropic" microstructure was generated when some melts were rapidly solidified by immersing into water a mould that contains the melts. In this study, the rapid cooling rate was achieved by solidifying the ingots under pressure. This approach used in this study has been patented by the author and his co-worker, and is an easy and feasible way to produce the IM hypoeutectic Al-Si thixocasting feedstock billets [110].

5.2.2 T6 Heat treatment

T6 thermal treatment was applied to strengthen these specimens. The T6 treatment condition used for Al-25SiCuMg (PT) and (IT) alloys was 500°C for 4 hours followed by 175°C for 10 hours and it for LM13 alloys was 520°C for four hours, followed by 175°C for eight hours; whereas, Al-20Si-5Fe (PT) is an non-heat-treatable alloy.

5.2.3 Properties examination

A. Microstructural observation

Microstructure of the thixocast products was investigated by optical and scanning electron microscopes. The specimens were ground and polished and subsequently etched with a 0.5%HF water solution.

B. Tensile test

Tensile test samples were machined from the part of "Plate" in the specimen shown in Fig. 4.13 a. The dimensions of the tensile specimens are shown in Fig. 4.13 b. The tests were conducted on an Instron 4469 machine at a strain rate of $1.1\times10^{-3}\ s^{-1}$. The data for each condition were obtained from the measurements of 3 specimens.

The tensile fracture surfaces were examined using a Cambridge S-360 Scanning electron microscope SEM.

C. High temperature compression

Compression strength at elevated temperature was conducted using a Gleeble-2000 system thermo mechanical simulator and using cylindrical specimens of 8mm in diameter and 10 mm in height. The specimens were machined from the part of "Runner" in the specimen shown in Fig. 4.11. The compression strain rate was $0.1\ s^{-1}$.

5.3 Results and Discussions

5.3.1 Microstructure

A. Powder-thixocast Al-Si-Cu-Mg and Al-Si-Fe alloys

Figures 5.2 show the optical micrographs of Al-25Si-2.5Cu-1Mg powder before and after thixocasting and after T6 heat-treatment.

Figure 5.2 a shows Al-25SiCuMg powder contains fine and equiaxed particulates of primary Si phase that are uniformly embedded in a matrix. The matrix is composed mostly of α-Al grains and eutectic Si particles, which may be considered to be similar with the Al-12wt%Si eutectic formed under equilibrium conditions. A small amount of eutectic Si, $CuAl_2$ and $Al_5Cu_2Mg_8Si_6$ ('Q' [104]) may also exist to construct a black network in the matrix. The black network forms the boundaries of α-Al grains. After thixocasting, Fig. 5.2 b and c clearly reveals that primary Si particles of Al-25SiCuMg alloy have a tendency to cluster together. After T6 treatment, the black network in as-thixocast Al-25SiCuMg (PT) alloy almost disappeared (Fig. 5.2 d). Besides, the T6-treated alloy seems to exhibits no sign of eutectic Si particles (Fig. 5.2 d), although the Si content of this alloy exceeds the eutectic point (~12%).

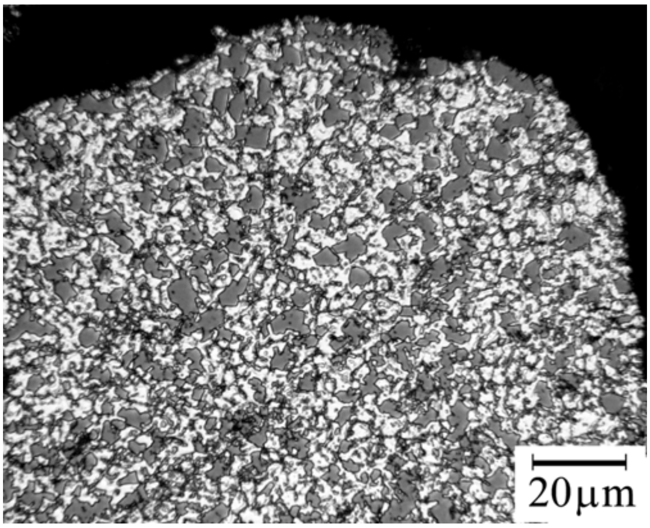
Figure 5.3 shows the optical micrographs of Al-20Si-5Fe powder before and after thixocasting. Figure 5.3 a shows that the Al-20Si-5Fe powders possesses long needle-shaped iron-rich particulates having size of 20-80μm, embedded in an Al-12%Si matrix. No black network was found in Al-20Si-5Fe powder. Figures 5.3 b and c shows that clustering of primary Si particles in Al-20SiFe alloy after thixocasting seems to be hampered by rod-shaped intermetallic, resulting in more uniform distribution of primary Si particles than that in Al-25SiCuMg (PT).

After thixocasting, the long needle-shaped intermetallic phases in the Al-20SiFe powder (Fig. 5.3 a) transformed to rectangular shape (Fig. 5.3 b and c). The needlelike phase is verified to be composed most of metastable δ-Al_4FeSi_2 of tetragonal crystal structure [106-109], whereas the rectangular phase is the equilibrium β-Al_5SiFe. The formation of non-equilibriumδ-Al_4Si_2Fe phase indicates that the powders undergo extensive undercooling, prior to solidification [106-109]. However, our results indicate that after thixocasting the metastable δ-Al_4Si_2Fe phase transformed to equilibrium β-Al_5SiFe phase, based on the X-ray diffration. It is suggested that the phase transformation of the δ-Al_4Si_2Fe particulates into β- Al_5SiFe phase and grain growth simultaneously occurred when a semisolid powder slurry was heated and deformed to fill a mold cavity.

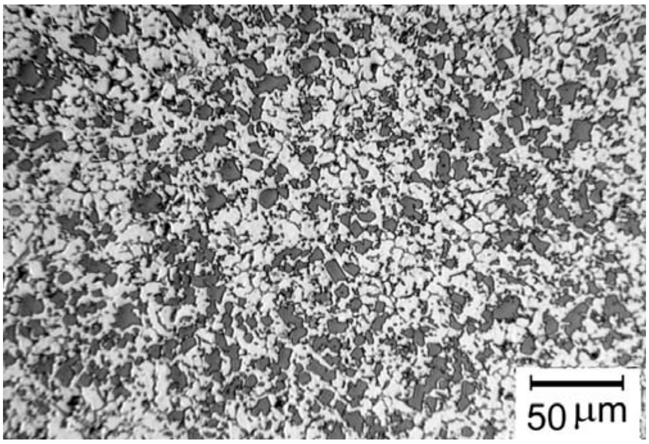
After thixocasting, the size of primary Si particles in as-atomized powders becomes larger. This microstructure coarsening is apparently due to semisolid processing of powders during consolidating and thixocasting. But, it is still worthy to note that the eutectic Si particulars in the matrix of Al-20SiFe alloy (Fig. 5.3 b) are much coarser compared with Al-25SiCuMg (Fig. 5.2 b). For example, the primary Si size in both powders is 3-9μm (Figs. 5.2 a, 5.3 a), whereas after thixocasting it becomes 5-13μm and 8-25μm in Al-25SiCuMg and Al-20SiFe, respectively (Figs. 5.2 b, 5.3 b).

This coarser eutectic matrix microstructure (Fig. 5.3 c) may indicate that the eutectic matrix in Al-20SiFe powder could once melted, leading to high liquid fraction after heating to semisolid state. Afterwards, when the powder was subsequently consolidated in a mold cavity, the melt was solidified at a low cooling rate, so that a coarser microstructure of eutectic matrix formed.

As mentioned in Chapter 4, we found that the coarsening behavior of primary Si particles could be retarded by stabilization of solid α-Al grains in the semisolid slurry. This is because that the solid α-Al grains in the slurry are considered can prevent primary Si particles from contacting with the melt, leading to low diffusion behavior and consequent to low Si grain growth. Since the Al-12%Si matrix of Al-20SiFe powder was almost liquefied during thixocasting, high fraction of liquid occurs and α-Al grains could hardly be stabilized in semisolid state. As a result, primary Si particle size in Al-20SiFe alloy becomes larger after thixocasting compared with Al-25SiCuMg alloy.

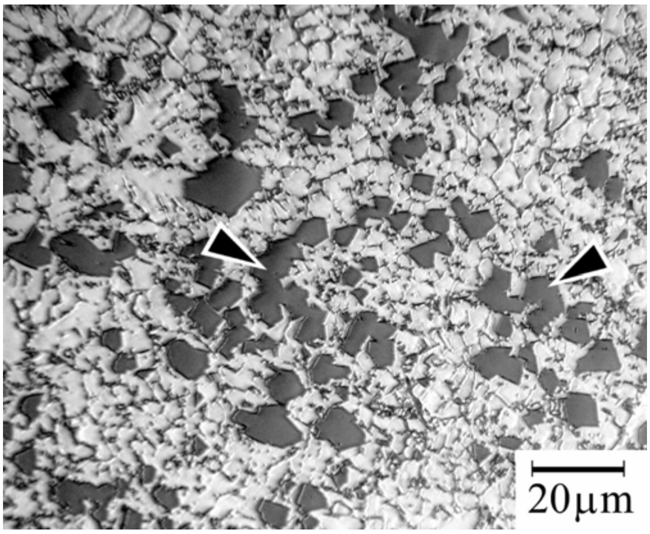


(a)

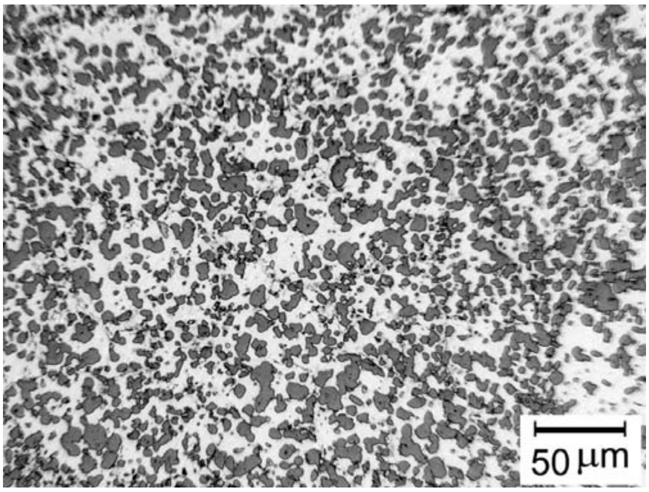


(b)

Figure 5.2 (continued)



(c)



(d)

Figure 5.2 Typical optical micrographs of Al-25Si-2.5Cu-1Mg alloy

(a) gas-atomized powder, (b) after powder-thixocasting, (c) a photograph magnified from (b), arrows indicate clustering of primary Si after thixocasting, and (d) the powder-thixocast alloy after T6 treatment.

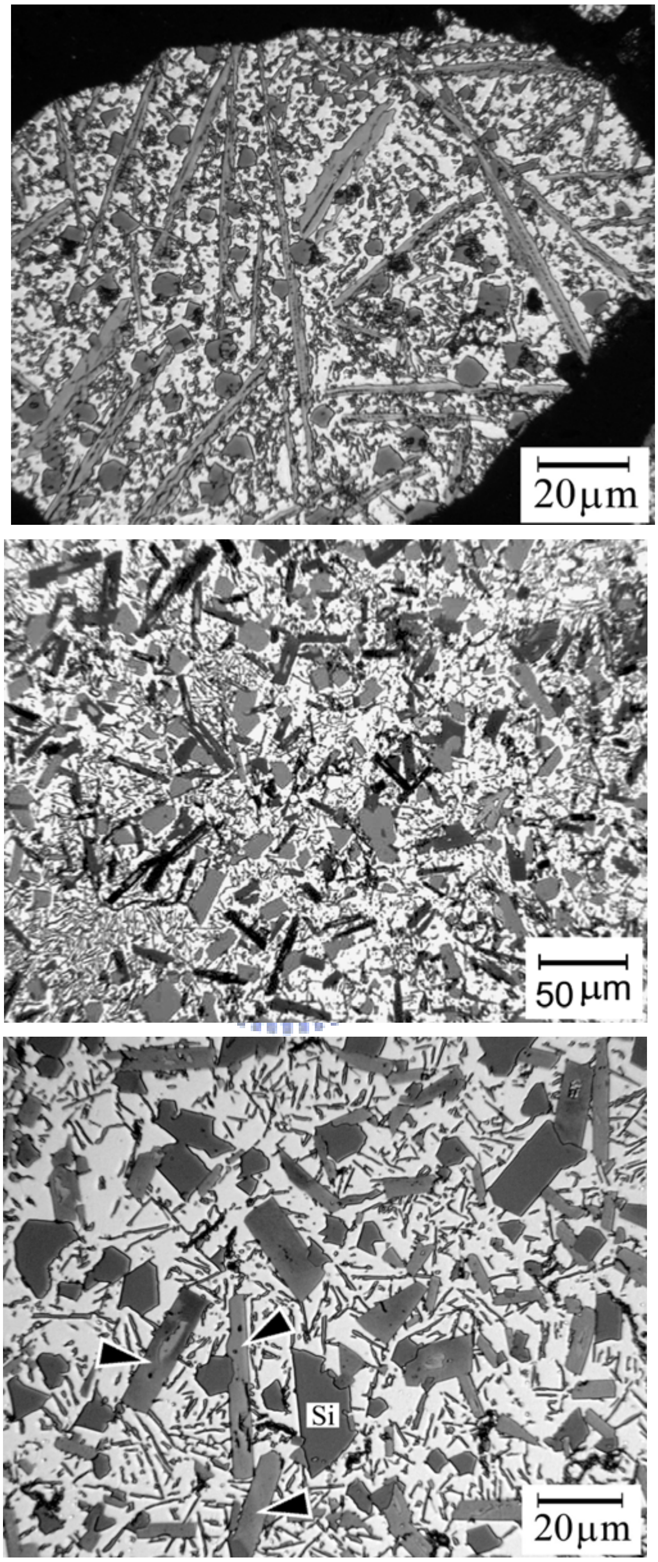


Figure 5.3 Typical optical micrographs of Al-20Si-5Fe alloy
(a) gas-atomized powder, (b) after powder-thixocasting, and (c) a photograph magnified from (b), arrows indicate β- Al_5SiFe phase in rod shape.

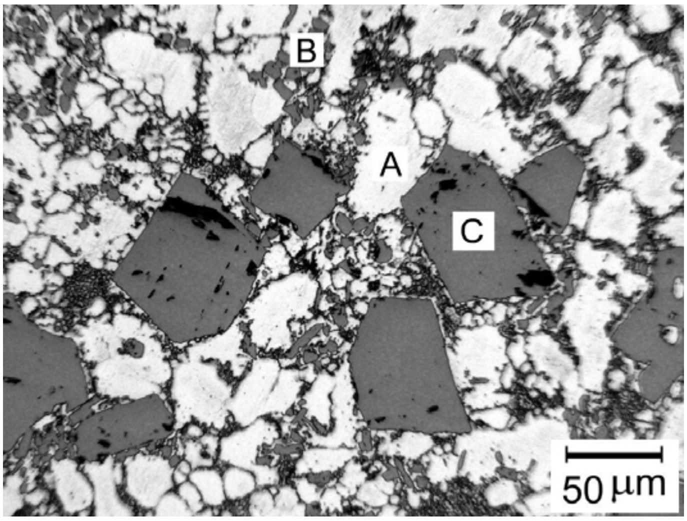
B. Conventional Al- Si-Cu-Mg alloys

Figures 5.4 and 5.5 present the optical microstructures of the as-prepared and T6-treated conventional Al-Si-Cu-Mg alloys, respectively.

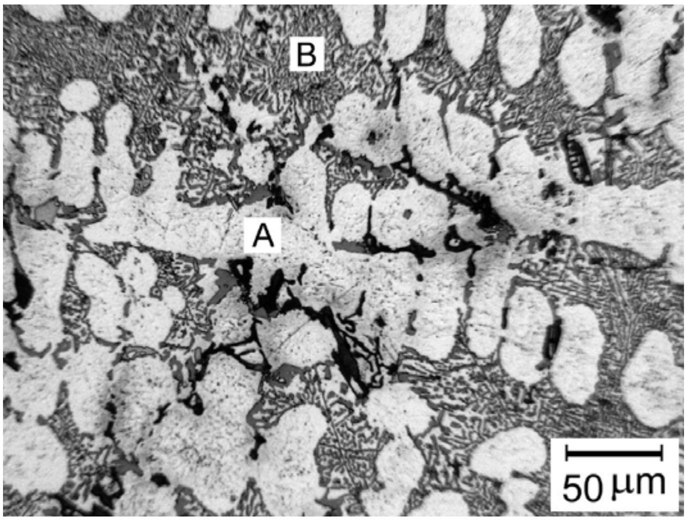
Al-25SiCuMg (IT) alloy contains coarse primary Si particles distributed in a matrix that comprises globular α-Al grains and polygonal eutectic Si particles (Fig. 5.4 a).   LM13 alloy has the typical microstructure of an Al-12wt%Si eutectic alloy that includes dendritic α-Al grains and fine needle-like eutectic Si particles (Fig. 5.4 b).

From comparing Fig. 5.5 with Fig. 5.4, it can be seen that after T6 treatment the primary Si particles of the two alloys become more spherical and round; meanwhile, the fiber-like eutectic Si particles in LM13 matrix changed into small equiaxed particles.

From comparing Fig. 5.4 with Fig. 5.2, Al-25SiCuMg (IT) alloy is found to have much coarse primary Si particles than does conventional Al-25SiCuMg (PT) alloy (approximately 100 µm against 8 µm).

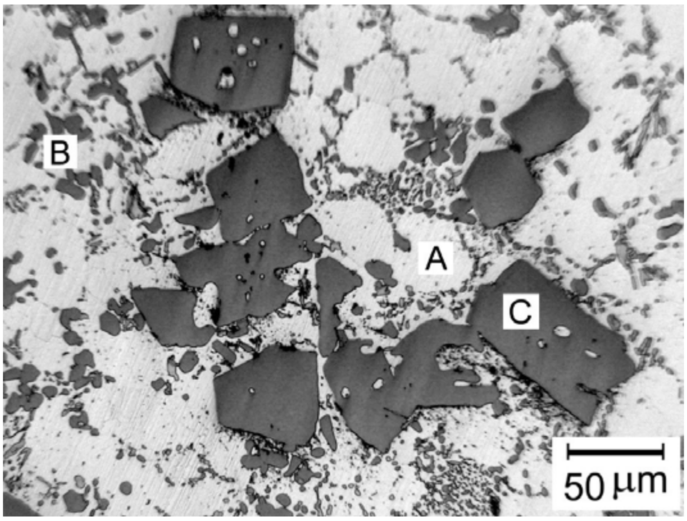


(a)

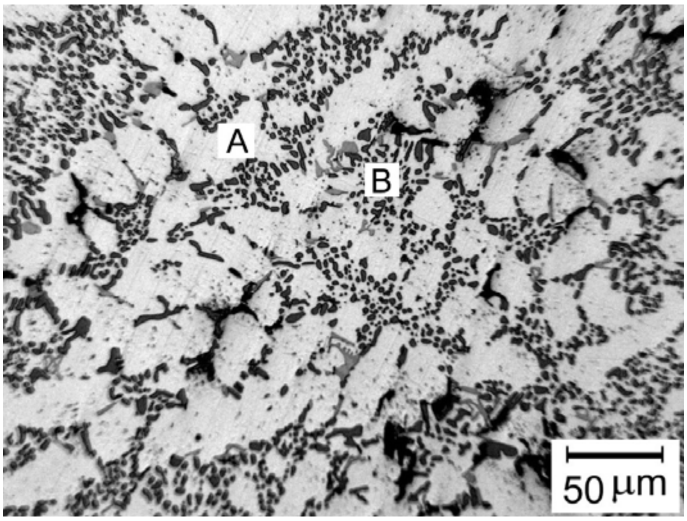


(b)

Figure 5.4 Optical microstructures of as-prepared conventional Al-Si alloys. (a) Al-25SiCuMg (IT), and (b) LM13. [A: α-aluminum; B: eutectic silicon particles; and C: primary silicon particles.]



(a)



(b)

Figure 5.5 Optical microstructures of T6-treated conventional Al-Si alloys. (a) Al-25SiCuMg (IT), and (b) LM13 alloys. [A: α-aluminum; B: eutectic silicon particles; and C: primary silicon particles.]