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Effects of Mn content on microstructures and mechanical properties of Al−5.0Cu−0.5Fe alloys prepared by squeeze casting

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Abstract: The effects of Mn content on the microstructures and mechanical properties of squeeze cast Al−5.0Cu−0.5Fe alloys at different applied pressures were examined by tensile test, optical microscopy, scanning electron microscopy and image analysis. The results show that the needle-like *β*-Fe phase (Al_7Cu_2Fe) is completely converted to the Chinese script *α*-Fe phase ($Al_{15}(FeMn)_3(CuSi)_2$) when the applied pressure is 0 MPa and the Mn/Fe mass ratio reaches 1.6. As to squeeze casting, the Mn/Fe mass ratio of 0.8 is demanded for the complete conversion of *β*-Fe phase to *α*-Fe phase at the applied pressure of 75 MPa. The lower Mn content, i.e., the less Mn/Fe mass ratio, for squeeze cast alloy is due to the small size and less content of the Fe-rich phases. Excessive amount of Mn, however, deteriorates the mechanical properties because of the increase in the total amount of *α*-Fe and the porosity that associates with the excessive brittle phases.

Key words: aluminum alloy; Mn; Fe-rich phase; squeeze casting; mechanical properties

1 Introduction

The heat treatable Al−Cu based alloys are widely used in the transportation, aerospace and military industry owing to their excellent mechanical and physical properties [1,2]. However, the solubility of iron in pure aluminum is very low (0.048%, mass fraction) and decreases by almost 5 times when 5% copper is added [3,4]. Almost all the iron will precipitate from liquid Al alloys in the form of Fe-rich intermetallic phases, mainly as the needle-like *β*-Fe phase. Therefore, the tolerance of Fe content is very poor, for example, in the 206 cast alloy family, the maximum iron content is usually limited to 0.15% (206.0 cast alloy) or less 0.10% (206.2 cast alloy) for general purpose use. In the aerospace applications, the Fe content is even required below 0.07% (A206.2) [5]. The low tolerance of Fe makes the alloys expensive to produce and limits their applications, especially with increasing use of the recycled metal charge.

In order to promote the utilization of recycled aluminum alloys, the alloys with high Fe content should be investigated. Many researchers studied the effect of Fe on the microstructures and properties in cast Al−Si alloys. It is found that the needle-like *β*-Fe phase is detrimental to the mechanical properties because it acts as stress raisers. Moreover, large Fe-rich needles tend to prevent the flow of liquid metal through the feeding channels and may cause porosity [6,7]. Mn addition is used to reduce the detrimental effects of the Fe-rich phases by replacing it with the less detrimental Chinese script *α*-Fe phase, resulting in the improvement of mechanical properties [8−11]. Generally speaking, it is agreed that Mn increases the strength and ductility of Al alloys due to the reduction of the embrittling effect via the formation of the Chinese script phase. However, Mn addition increases the total amount of the (Fe+Mn)-containing intermetallic phases [12]. Furthermore, the amount of Mn for *β*-Fe modification is uncertain because it depends on the composition and cooling rate. HWANG et al [8] studied the effect of Mn on the microstructures and mechanical properties of Al−Si−Cu casting alloys containing 0.5% Fe (mass fraction). They concluded that the plate-like *β*-Fe phase is completely converted to the Chinese script *α*-Fe phase at a Mn/Fe mass ratio of 1.2.

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SALEM et al [13] found that high cooling rates and Mn addition are not able to totally prevent the formation of β-Al_5FeSi needles even at Mn/Fe mass ratio of 2 in Al−Si alloy containing 0.3% Fe. The occurrence of the porosity associated with these phases will also influence the tensile properties. In the meantime, the copper-containing intermetallic phases formed at a low temperature of ternary eutectic reaction will lead to the feeding difficulty of residual liquid [14,15].

Until present, little work on the microstructures and properties of Al−Cu based alloys with high Fe content has been reported, especially the relationship among the mechanical properties, the Fe-rich phases and the porosity. TSENG et al [10] reported that almost all the plate-like β-Fe phases are completely converted to the Chinese script α-Fe phase when 0.66% Mn is added into the A206 alloy with 0.30% Fe. But they did not study the relationship between the porosity and Mn addition. Recently, LIU et al [16] have found a new Fe-rich intermetallic phase $Al_3(FeMnCu)$ with needle-like morphology in Al−4.6Cu−0.5Fe−0.5Mn−0.1Si alloy at a cooling rate of 0.2 K/s. This new phase is different from the Chinese script $Al_3(FeMn)$ mentioned in the classic reference of MONDOLFO [17]. The results indicate that the Mn addition may not modify the morphology of the platelet-like intermetallic phases in 206 alloys, which is different from the well-known observations in the cast Al−Si alloys.

Squeeze casting is a method of producing near-net-shape components. The applied pressure increases the heat-transfer coefficient between the die and the castings and leads to the comparatively short solidification time, high cooling rate and fine-scale microstructures [18,19]. DONG et al [20] found that squeeze casting is more tolerant of Fe impurities in Al−7Si−0.3Mg alloys due to the formation of smaller Fe-rich phases. However, there is little work on the microstructures and mechanical properties of Al−Cu alloys with high Fe contents as a function of Mn addition in squeeze casting. Therefore, the effects of the Mn/Fe ratio on the microstructures and mechanical properties of squeeze cast Al−5.0Cu−0.5Fe alloys were studied in this work. And the microstructures of the alloys were focused on the intermetallic compounds, the α(Al) size and the porosity.

2 Experimental

Commercially pure Al (99.5%), Al−50%Cu, Al−10%Mn and Al−5%Fe master alloys were used to prepare the experimental alloy and the final chemical compositions were analyzed using an optical emission spectrometer, as shown in Table 1. The raw materials were melted in a crucible furnace at 1053 K, and then the melt was poured into a cylindrical die under different applied pressures from 0 to 75 MPa. The die temperature was set at approximately 523 K, and the melt temperature was set at 983 K before squeeze casting. Finally, the samples with the sizes of 65 mm in height and 68 mm in diameter were obtained.

Table 1 Chemical compositions of experimental alloys

Alloy	Mass fraction/%					Mn/Fe mass ratio
	Cu	Mn	Fe	Si	Al	
Al−5.0Cu−0.4Mn−0.5Fe (Mn04)	4.89	0.39	0.46	0.07	Bal.	0.8
Al−5.0Cu−0.6Mn−0.5Fe (Mn06)	4.92	0.59	0.46	0.08	Bal.	1.2
Al−5.0Cu−0.8Mn−0.5Fe (Mn08)	5.07	0.78	0.47	0.07	Bal.	1.6
Al−5.0Cu−1.0Mn−0.5Fe (Mn10)	5.00	0.93	0.45	0.07	Bal.	2.0

All samples for tensile test were cut into the dimensions of d 10 mm×65 mm by line-cutting machine from the same radius of the castings. The test bars were pulled to fracture at room temperature using SANS CMT5105 standard testing machine and the data were an average value from at least three tested samples. The samples for the metallographic observation were cut in the gauge length apart from selected tensile specimens. The metallographic samples were etched with 0.5% HF solution for 30 s to reveal the microstructures. A Leica light optical microscope equipped with the image analysis software was used to study the microstructures, the average volume fraction and the size of intermetallic phases. To get statistically significant data from the image analysis, approximately 30 different regions were analyzed. The area fraction of the porosity was determined by analyzing metallographic sections of tensile specimens with a Leica light optical microscope and a scanning electron microscope of Nova Nano SEM 430. In order to reveal the relationship between the porosity and Mn/Fe mass ratio of the alloys, the density and porosity values were also calculated and measured according to the ASTM standard D3800 and C948 [21]. The average compositions of various phases were analyzed using Nova Nano SEM 430 equipped with an energy-dispersive X-ray analyzer (EDX). The results reported were an average of five spot analyses.

3 Results

3.1 Mechanical properties

Figure 1 shows the mechanical properties of the as-cast alloys at different applied pressures and Mn contents. When the applied pressure is 0 MPa, the ultimate tensile strength (UTS), yield strength (YS) and elongation increase with Mn content up to 0.8% and then decrease distinctly. However, when the applied pressure is over 25 MPa, the UTS increases with Mn content up to 0.6% and then decreases slightly. As to the elongation of the alloys, there is a slightly reduction with the increase of Mn content. It is also found that all the UTS, YS and elongation of the four alloys increase as the applied pressure increases from 0 to 75 MPa.

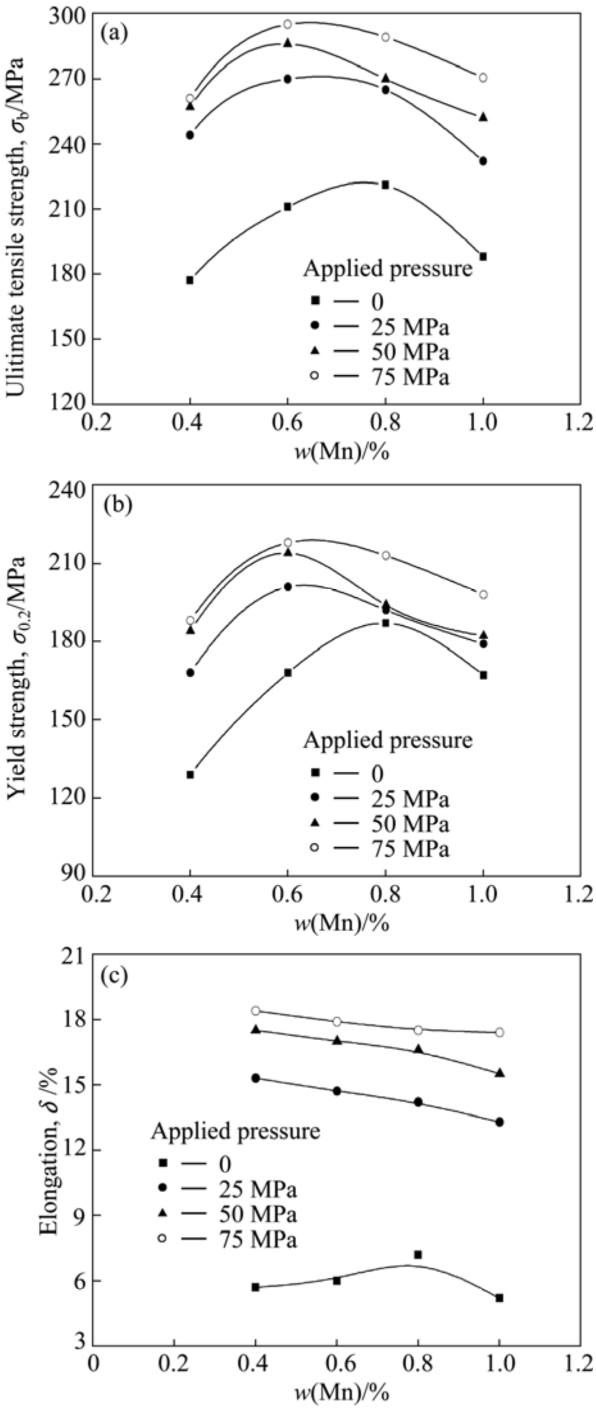

Fig. 1 Ultimate tensile strength (a), yield strength (b) and elongation (c) of as-cast alloys varying with Mn content at different applied pressures

3.2 Microstructures

Figure 2 shows the microstructures of the as-cast Mn06 alloy at different applied pressures. The second dendritic arm spacing of α(Al) decreases distinctly and the porosity is hardly detectable with the increase of the applied pressure. The microstructures of the four different samples consist of α(Al) dendrites, Fe-rich phases (needle-like and Chinese script) and copper-containing intermetallic phase as shown in Fig. 3. The EDS results show that the copper-containing intermetallic phase (marked with C in Fig. 3) contains 35.94% Cu, which is in agreement with the stoichiometry of $\theta(Al_2Cu)$. The average compositions of the Fe-rich phases (marked with A and B in Fig. 3) measured by EDS analysis are given in Table 2. The results show that the composition of the Chinese script Fe-rich phase observed in Mn04 to Mn10 alloys has similar mole ratio of n(Al):n(Fe+Mn):n(Cu+Si) of approximately 15:3:2, which is α-Fe ($Al_{15}(FeMn)_3$-$(SiCu)_2$) according to LIU's study [3]. It should be noted that the Mn/Fe mole ratio in the α-Fe phase increases distinctly from 0.28 in the Mn04 alloy to 0.69 in the Mn10 alloy.

When the applied pressure is 0 MPa, it is found that there are some needle-like β-Fe phases present in Mn04 and Mn06 alloys, indicating that the Mn concentration is insufficient to eliminate the β-Fe phase completely. However, the needle-like β-Fe phase is almost undetectable in Mn08 alloy, which means that the needle-like β-Fe phase is almost completely converted to the Chinese script α-Fe phase when the Mn content is up to an amount corresponding to the Mn/Fe mass ratio of 1.6. In contrast, a Mn/Fe mass ratio of 0.8 is enough to convert the needle-like β-Fe phase to the Chinese script α-Fe phase when the applied pressure increases to 75 MPa. Meanwhile, with the increase of applied pressure, all the Fe-rich phases become smaller compared with those of the alloys at 0 MPa.

However, excessively high Mn content beyond the need to eliminate the β-Fe phase will lead to greater area fraction of the α-Fe phase, as shown in Table 3, in which the measured area fractions of the second phases are summarized. It can be found that the area fraction of the α-Fe phase in the alloys increases while the amount of θ phase decreases slightly with increasing the Mn content when the applied pressure is 0 MPa. However, all the amounts of the second phases decrease distinctly when the applied pressure increases to 75 MPa. Since higher volume fraction of the α-Fe phase is also detrimental to the alloy's properties, it is necessary to control the Mn/Fe ratio so that the complete-elimination of β-Fe phase and the minimization of the amount of α-Fe phase can be balanced.

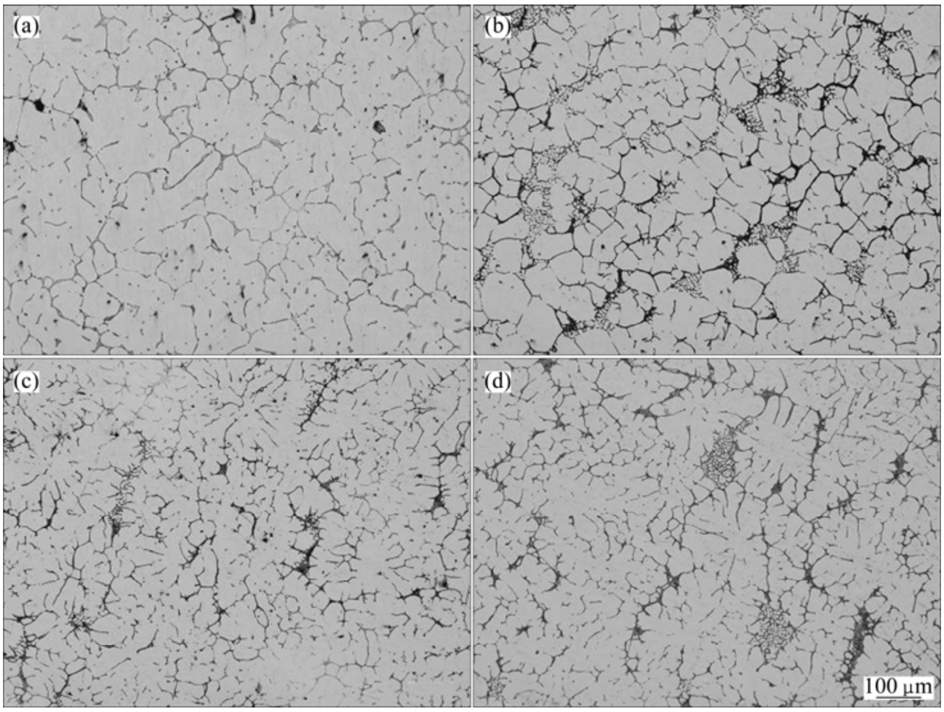

Fig. 2 Microstructures of as-cast Mn06 alloy at different applied pressures: (a) 0 MPa; (b) 25 MPa; (c) 50 MPa; (d) 75 MPa

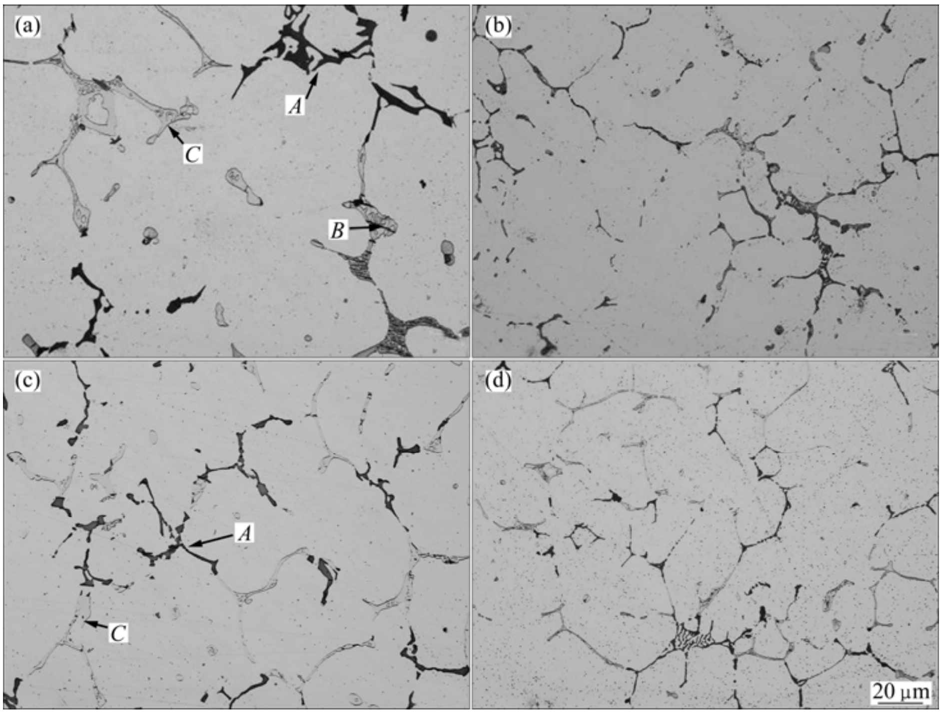

Fig. 3 Optical microstructures of as-cast alloys with different Mn contents and applied pressures: (a) Mn04, 0 MPa; (b) Mn04, 75 MPa; (c) Mn10, 0 MPa; (d) Mn10, 75 MPa

Table 2 Average compositions of Fe-rich phases

Alloy	Location in Fig. 3	Mole fraction/%					Phase
		Al	Cu	Mn	Fe	Si	
Mn04	*A*	72.41	9.71	3.74	13.31	1.39	$Al_{15}(FeMn)_3(CuSi)_2$
	B	82.3	11.9	−	5.0	−	Al_7Cu_2Fe
Mn06	*A* (Figure not shown)	75.28	9.62	3.98	10.13	1.41	$Al_{15}(FeMn)_3(CuSi)_2$
	B (Figure not shown)	67.8	27.3	0.60	4.3	−	Al_7Cu_2Fe
Mn08	*A* (Figure not shown)	77.44	8.16	5.19	8.65	1.15	$Al_{15}(FeMn)_3(CuSi)_2$
Mn10	*A*	74.84	11.34	5.15	7.47	1.20	$Al_{15}(FeMn)_3(CuSi)_2$

Table 3 Area fractions of second phases

Alloy	Pressure	Area fraction/%			Total area fraction/%
		β-Fe	α-Fe	$\theta(Al_2Cu)$	
Mn04	0 MPa	0.4	1.9	4.2	6.5
	75 MPa	−	0.9	1.2	2.1
Mn06	0 MPa	0.2	2.3	3.5	6.0
	75 MPa	−	1.1	1.6	2.7
Mn08	0 MPa	−	2.5	3.3	5.8
	75 MPa	−	1.1	1.6	2.7
Mn10	0 MPa	−	2.9	3.1	6.0
	75 MPa	−	1.3	1.3	2.6

3.3 Porosity

The excess of Mn not only leads to the higher volume fraction of the α-Fe phase, but also results in the greater porosity associated with the second phases. Figure 4 shows the porosity associated with the β-Fe phase in the Mn04 alloy and the α-Fe phase in Mn10 alloy when the applied pressure is 0 MPa. The total area fraction of porosity observed in these samples was estimated from the metallographic cross sections. The porosity was measured using standard image analysis techniques, and the average values are given in Table 4.

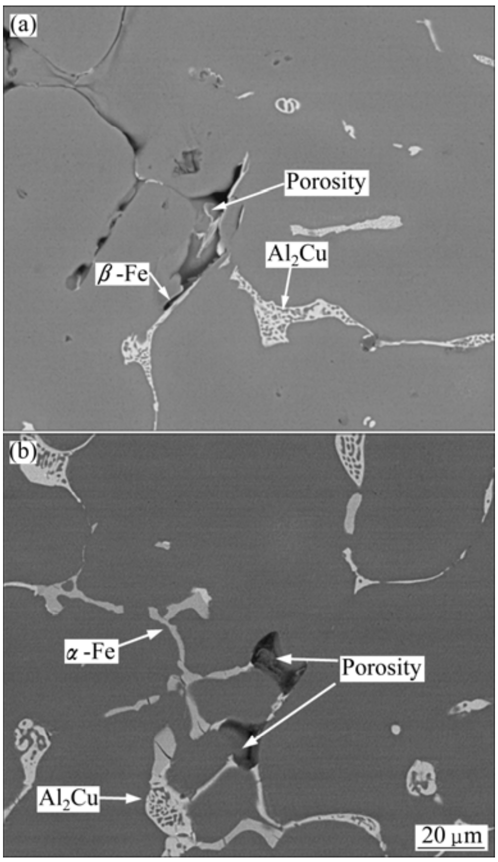

Fig. 4 SEM images showing porosity associated with β-Fe phase in Mn04 alloy (a) and α-Fe phase in Mn10 alloy (b) at applied pressure of 0 MPa

As shown in Table 4, the minimum area fraction of porosity is observed in the Mn08 alloy that is the optimum Mn content to completely eliminate the β-Fe phase and form the minimum amount of the α-Fe phase.

Table 4 Area fraction of porosity measured from metallographic surface of alloys at applied pressure of 0 MPa

Alloy	Area fraction of porosity/%
Mn04	0.20−0.28
Mn06	0.10−0.20
Mn08	0.06−0.18
Mn010	0.10−0.26

In order to reveal the relationship between the porosity and Mn/Fe ratio at different applied pressures, the density and porosity were calculated and tested. The theoretical densities of the alloys were calculated based on the actual compositions shown in Table 1. The actual density values of the alloys were measured by Archimedes method. The volume fraction of porosity (f) was calculated by the following equation (ASTM Standard C948) [26]:

$$f=\left[\frac{D_t-D_a}{D_t}\right]\times 100\% \tag{1}$$

where D_t and D_a are the theoretical and actual densities of the alloys, respectively. The volume fractions of porosities of the as-cast alloys are shown in Fig. 5. It can be found that the porosities are consistent with the results in Fig. 5. It seems that the Mn addition can improve the volume fraction of porosity slightly, the volume fraction of porosity decreases from 3.80% in Mn04 alloy to 2.82% in Mn08 alloy. Furthermore, the applied pressure decreases the volume fraction of porosity distinctly. The volume fraction of porosity decreases by 50%−70% in all the alloys when the applied pressure is 75 MPa.

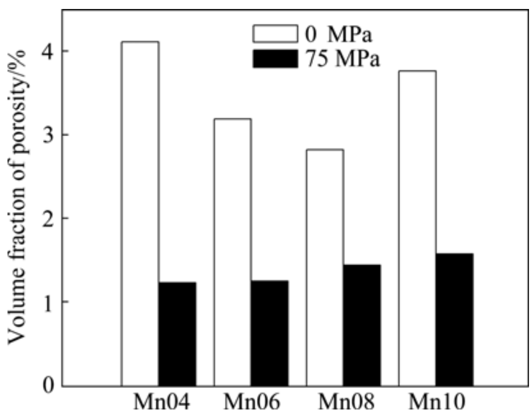

Fig. 5 Volume fractions of porosities of as-cast alloys at different applied pressures

4 Discussion

4.1 Effect of Mn/Fe ratio on porosity

Porosity is generally easy to form in the Al−Cu alloy due to its large freezing range. If impurity Fe is added to the Al−Cu alloys, the porosity becomes worse due to the impeding effect of iron platelets on the flow of liquid metal during the solidification and the lack of sufficient amount of eutectic liquid at the end of solidification process. As shown in Figs. 5 and 6, Mn addition can improve the porosity slightly. However, the excess of Mn leads to the greater porosity. The reason is that the Mn/Fe ratio affects not only the amount of (Fe+Mn)-containing intermetallic compounds but also other intermetallic phases, such as $\theta(Al_2Cu)$ phase. As shown in Table 3, the amount of θ phases decreases slightly with the increase of Mn content. The $\theta(Al_2Cu)$ formed in ternary eutectic reaction at very low temperature (538 °C) often leads to the difficulty of residual liquid feeding. Furthermore, the occurrence of the needle-like β-Fe phases in Mn04 alloy indicates that the Mn/Fe mass ratio of 0.8 is insufficient to completely convert the β-Fe phase. Some results in Al−Si alloys show that needle-like Fe-rich phases tend to prevent the flow of liquid metal through the feeding channels and may cause porosity [6,7]. Therefore, the minimum porosity is observed in the Mn08 alloy, which is consistent with the Mn/Fe mass ratio of 1.6 necessary to completely eliminate the β-Fe phase and form the minimum amount of the α-Fe phase. Further increase of the Mn/Fe mass ratio to 2.0 leads to higher amount of excessive α-Fe phase, which associates with the large porosity as shown in Fig. 4(b).

When the pressure is applied to the melt during squeeze casting, it is found that the porosity percentage in the alloys decrease distinctly and the porosity is hardly observed. This is attributed to the improvement of fluidity of the liquid metal in solidification under high pressure. In the meantime, the significant decrease of $\theta(Al_2Cu)$ content and the refinement of Fe-rich phases are also helpful for the vanishing of the porosity in the squeeze casting alloys.

4.2 Effect of Mn/Fe ratio on mechanical properties

From the experimental results, it is found that there is an optimal Mn/Fe ratio for the best mechanical properties of the alloys. The optimal Mn/Fe mass ratios are 1.6 and 1.2 for the UTS and YS when the applied pressure is 0 MPa and 75 MPa, respectively. In Al−Cu alloys, the mechanical properties are generally affected by the grain size of α(Al), the second phases and the porosity. It has been proved in Al−Si alloys that the detrimental effects of the Fe-rich phase can be lightened by replacing β-Fe phase with α-Fe phase. In this work, it is also confirmed that the increase in UTS with Mn addition is clearly related to the decrease in the amount of the needle-like β-Fe phase. The Mn/Fe mass ratio of 1.6 is just correlated to the complete conversion of β-Fe phase to α-Fe phase when the alloy is prepared without pressure.

When the pressure of 75 MPa is applied in squeeze casting, the β-Fe phase is easily converted to the Chinese script α-Fe phase completely with the Mn/Fe mass ratio of 0.8 (Fig. 3 and Table 3). The less Mn/Fe mass ratio of the squeeze cast alloys is due to the small size and low content of the Fe-rich phases caused by the high cooling rate. It is proved that the higher cooling rate can be achieved during the solidification in squeeze casting. It is also in accordance with the results of KAMGA et al [4], who pointed out that the high cooling rate was essential to prevent the formation of the β-Fe phase. MAENG et al [22] also reported that the length of the Chinese script Fe-rich phase decreased with increasing the applied pressure probably because the higher solidification rate impeded the growth of the Fe-rich phases. Therefore, less Mn is neccesary for the complete replacement of the β-Fe phase with the α-Fe phase in squeeze casting.

It should be noted that the Mn/Fe mass ratio is 1.2 for the highest UTS and YS of the alloys in squeeze casting. From Table 3, it can be seen that the amount of all the second phases decrease distinctly when the applied pressure is 75 MPa. This means that more second phases are required to strengthen the alloy although the needle-like β-Fe phase has already converted into the Chinese script α-Fe phase completely. Hence, the Mn content of 0.6% is still useful for the strength of the squeeze cast alloy.

5 Conclusions

1) When the applied pressure is 0 MPa and the Mn/Fe mass ratio is 1.6, the needle-like β-Fe (Al_7Cu_2Fe) phase is completely converted to the Chinese script α-Fe ($Al_{15}(FeMn)_3(CuSi)_2$) phase in the Al−0.5Cu−0.5Fe alloys.

2) Only Mn/Fe mass ratio of 0.8 is needed for the complete conversion of β-Fe phase to α-Fe phase when the applied pressure is 75 MPa. The less Mn/Fe ratio for squeeze cast alloy is due to the small size and lower content of the Fe-rich phases.

3) Excessive amount of Mn will deteriorate the mechanical properties of the alloys because it will increase the total amount of α-Fe phase and the porosity.

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Mn 含量对挤压铸造 Al−5.0Cu−0.5Fe 合金显微组织和力学性能的影响

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摘 要：采用拉伸性能测试、光学显微镜、扫描电镜和定量金相测试手段研究 Mn 含量对不同压力下挤压铸造 Al−5.0Cu−0.5Fe 合金显微组织和力学性能的影响。结果表明：当挤压压力为 0 MPa，Mn/Fe 质量比达到 1.6 时才能将针状β-Fe 相(Al_7Cu_2Fe)完全转变成汉字状α-Fe 相($Al_{15}(FeMn)_3(CuSi)_2$)。而对于挤压铸造，当挤压压力为 75 MPa 时，在 Mn/Fe 质量比为 0.8 时就可以将β-Fe 相完全转变成α-Fe 相。挤压铸造合金中需要的 Mn 含量较低，即 Mn/Fe 质量比较小，这主要是由于在挤压压力下富 Fe 相的细化以及相比例的减少。然而，加入过量的 Mn 将导致合金力学性能的下降，这是因为过量的 Mn 将导致α-Fe 相的增多及这些多余的硬脆相导致的孔洞增多。

关键词：铝合金；Mn；富铁相；挤压铸造；力学性能

(Edited by Wei-ping CHEN)