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ARTICLE

Synergistic Effect of Sr and Ca Combined Additions on the Microstructure of A390 Aluminum Alloy

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Abstract: Effects of Sr and Ca combined additions (Sr+Ca=0.05wt/%) on the microstructure of A390 aluminum alloy were evaluated by investigating the variation of quantity, size and shape of primary silicon, eutectic silicon, primary α phase and intermetallic phase. The results show that the variation of microstructures results from the synergistic effect of Sr-Ca additions. The loss rate of Sr is reduced with a small amount of Ca; meanwhile, the coarsening effect of primary silicon phase arising from Sr addition is also suppressed. The Sr in A390 aluminum alloy mainly modifies eutectic silicon phase and refines θ phase. Furthermore, Ca principally modifies primary silicon phase and primary α phase. The optimal microstructure is obtained when combined addition of 0.05%Sr-Ca is applied and the atomic ratio of Sr and Ca is 5:1, in which primary silicon phase and eutectic silicon phase as well as primary α phase are all modified significantly compared with those of only 0.05% Sr addition.

Key words: Al-Si alloys; modification; grain refinement; microstructure

A390 aluminum alloy is typical hypereutectic Al-Si alloy with 16%~18% silicon content. It has been widely used in auto-motive due to its excellent properties, including good abrasion and corrosion resistance, low coefficient of thermal expansion and high strength-to-weight ratio[1,2]. However, its microstructure mainly consists of coarse bulk primary silicon phase, long needle-like eutectic silicon phase and dendritic primary α phase[3], which result the poor mechanical properties because coarse and needle-like silicon phase crystals lead to premature crack initiation and fracture in tension. Therefore, the microstructure of A390 aluminum alloy must be modified and refined to improve the mechanical properties and expand the applied range.

Sr is the most effective modification element to refine the microstructure of Al-Si alloy. Sr is generally used due to the features of long effective modification period, repeated remelting and no harmful gas. However, the chemical properties of Sr are particularly active, and it is easy to oxidize and burn out. Therefore, Al-10Sr master alloy is usually added to reduce the burning loss rate of Sr. The optimum addition amount is 0.3%~0.8% and the melting temperature is 654~770 °C[4,5]. Researchers have reported the modifying effect of Sr on the eutectic silicon phase and Fe-rich intermetallic phase[6-8]. Sui[6] investigated the influence of Sr content on Al-12Si eutectic silicon phase and primary α phase and concluded that the best modification effect could be obtained when the content of Sr was 0.02%. Under this content, Sr induced undercooling before solidification. The eutectic silicon phase was transformed from needle-like to fiber, and the primary α phase was refined. The results of J. Espinoza-Cuadra[7] show that the impurity of Al_5FeSi phase in A319 industrial alloys could be modified from platelets or needle-like to Chinese script-like shape by Sr addition. Timple[8] further studied the influence of holding time on eutectic silicon phase during modification, and found that the fibrous eutectic obtained by modification of 5 min was lost after 60 min because of Sr oxidation during prolonged melt holding. In order to achieve a better modification effect of Sr, it is used in combination with other elements, such as Sr-Mn, Sr-AlTiB, Sr-B, Sr-Sb,

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Sr-RE and Sr-P[9-19]. Kumari[9] reported that Sr-Mn combined addition had a modification effect on eutectic silicon phase and Fe-rich phase in Al-7Si-0.3Mg-0.8Fe aluminum alloy. Li[10] and Lu[11] studied the effect of combined addition of Si-AlTiB in hypoeutectic and near-eutectic Al-Si alloys. The results show that Si-AlTiB addition can refine both primary *α* phase and eutectic Si phase. In contrast, Liao[12] found that B can result in a serious poisoning effect on Sr, given that the chemical reaction of Sr and B generated SrB_6 compounds which inhibit the modification of Sr. And the result was supported by Samuel[13]. Qiu[14] presented that the A356.2 aluminum alloy with 0.5% Al-6Sr-7La exhibited optimal microstructure and mechanical properties, and the secondary dendrite arm spacing decreased to 17.9 μm and acicular-like eutectic silicon phase transformed to fibrous. Till now, many studies reported in the literature have focused on the Sr modification of eutectic silicon phase and intermetallic compound in hypoeutectic and near-eutectic Al-Si alloys, while few studies have been carried out on the Sr modification of primary silicon phase. Tebib[17] reported that Sr-P combined addition reduced the eutectic temperature, and therefore increased the number density of Mg_2Si particles, Si and π-Fe phases in the alloy. However, the most common modifier P for primary silicon phase had the characteristics of high burning rate, low ignition and producing P_2O_5 toxic gas during smelting, which can be replaced effectively by Ca. In addition, Ca also has the advantages of slow deterioration and no incubation period, and it has shown effective modification to primary silicon phase and eutectic silicon phase[18,19]. However, the effect of Sr-Ca composite modification on the microstructure of Al-Si alloys has not been realized yet.

In the present work, microstructure of A390 aluminum alloy modified by Sr-Ca composite addition was investigated. The variation of quantity, size and shape of primary silicon, eutectic silicon and primary *α* phase as well as intermetallic phase was intensively studied. Nucleation and growth mechanism of silicon phase metamorphism was also discussed.

1 Experiment

Unmodified A390 aluminum alloy given in Table 1 was selected as base alloy in order to achieve a microstructure with primary phase and eutectic phase. Sr and Ca were added in the form of Al-10Sr and Al-7.5Ca master alloy, respectively, for modification and grain refining.

The smelting trial was carried out in a graphite crucible resistance furnace equipped with KSW-5-12A type temperature measuring controller. Detailed steps were as follows: Firstly, preheating the graphite crucible to 400 °C, adding 3 kg A390 alloy until complete melting. Secondly, pressing the Al-10Sr and Al-7.5Ca master alloy at 720 °C. The atomic ratio of Sr and Ca was changed, while the total amount of Sr and Ca maintained 0.05%, as shown in Table 2. Thirdly, adding refining agent and holding for 10 min. Finally, pouring the molten aluminum into a metal mould preheated for half an hour at 200 °C. The die cavity size is *Φ*20 mm×30 mm. The specimens for component inspection and microstructure analysis were taken from ingot bottom 10 mm.

The aluminum oxide on the surface of the samples was removed by a lathe, and then its chemical composition was meas-ured by optical emission spectrometer (OES, ARL3460) according to GB/T 7999-2007. The average value of the 3 measurements was taken as the final value. The microstructure was observed with a metallurgical microscope (OM, OLYMPUS- DM2000) and a scanning electronic microscope (SEM, EVO-48). The chemical composition of the phases was detected with an energy dispersive spectrometer (EDS, Oxford), as shown in Table 3. The surfaces of the samples used for the observation were polished and etched with a 0.5% HF solution agent according to GB/T 3246-2000. The factors related to quantity, size and shape of primary silicon, eutectic silicon and primary *α* phase were calculated by quantitative analysis software (Image-Pro Plus 6.0) against 30 fields according to GB/T 15749-2008. The average value was taken as the final experimental value.

Table 1 Chemical composition of A390 aluminum alloy(wt%)

Si	Cu	Mg	Fe	Zn	Ti	Mn	Sr	Ca	Al
16.5	4.8	0.52	0.16	0.036	0.0125	0.0079	0.0025	0.0010	Bal.

Table 2 Content of Sr and Ca in A390 aluminum alloy

Sample	Total amount of Sr and Ca/wt%	$n_{Sr}:n_{Ca}$
A	-	-
B	0.05 Sr	-
C	0.05 Sr-Ca	5:1
D	0.05 Sr-Ca	2.5:1
E	0.05 Sr-Ca	1:1
F	0.05 Sr-Ca	1:2.5

2 Results and Discussion

2.1 Effect of Sr-Ca combined addition on composition and Sr loss rate

Fig.1 shows the contents of Sr and Ca in each sample of A390 aluminum alloy. It can be noticed that with the decrease of Sr and increase of Ca, the content of Sr shows an downward trend after first rising, and the content of Ca has an upward tendency. When the atomic ratio of Sr and Ca is 5:1,

Table 3 Chemical composition of the phases observed in present alloys

Figure No.	Atom fraction/%	Calculated formula	Suggested phase[20]
3f	Si: 99.5 Al: 0.5 Total: 100	Si	Primary silicon
5e	Al: 68.5 Si: 30.9 Cu: 0.04 Total: 99.8	Si+α-Al+traces of Cu	Eutectic silicon
5f	Al: 99.7 Si: 1.6 Cu: 0.05 Total: 99.8	α-Al+traces of Cu	Primary α
8e	Al: 68.7 Cu: 31.2 Total: 99.9	$CuAl_2$	θ
8f	Al: 66.5 Si: 20.6 Fe: 12.6 Total: 99.7	Al_5FeSi	β-Fe

the content of Sr reaches the highest value of 0.0385%, meanwhile the content of Ca is 0.0025%. Sr loss rate with different Sr and Ca atomic ratio is illustrated in Fig.2. The results show that Sr loss rate firstly decreases and then increases with the decrease in the Sr and Ca atomic ratio. The Sr loss rate is 58% with no Ca addition, since Sr is a chemically active element and reacts with water and oxygen to generate $Sr(OH)_2$ and SrO, respectively. When a small amount of Ca is added, a drastic decrease in the Sr loss rate (drops to a minimum of 13.6%) can be observed. It is to be noted that with the decrease in Sr and Ca atomic ratio, Sr loss rate increases, but it is lower than that of no Ca addition (sample B) invariably. It is shown that the addition of Ca has a beneficial effect on the decrease in Sr loss rate. This is due to the existence of Ca and Sr both belonging to the alkaline earth metals and Ca, which effectively consumes the water and oxygen in the melt, thereby preserving the Sr in the melt and reducing the burning loss rate of Sr.

2.2 Effect of Sr-Ca combined addition on primary silicon phase

Fig.3 shows the optical microstructure of primary silicon phase with different Sr and Ca additions in A390 aluminum

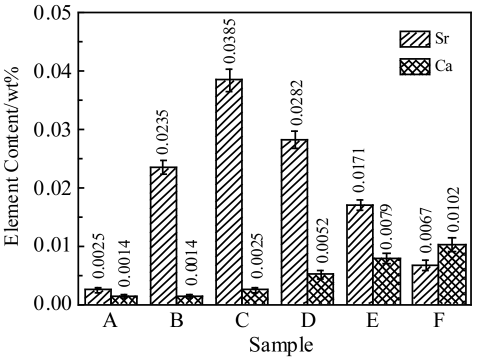

Fig.1 Contents of Sr and Ca in the A390 alloys

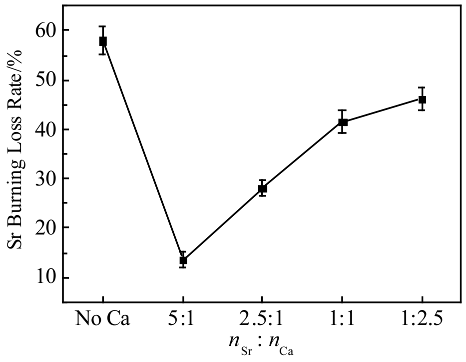

Fig.2 Sr loss rate of A390 alloy with different Sr and Ca molar ratios

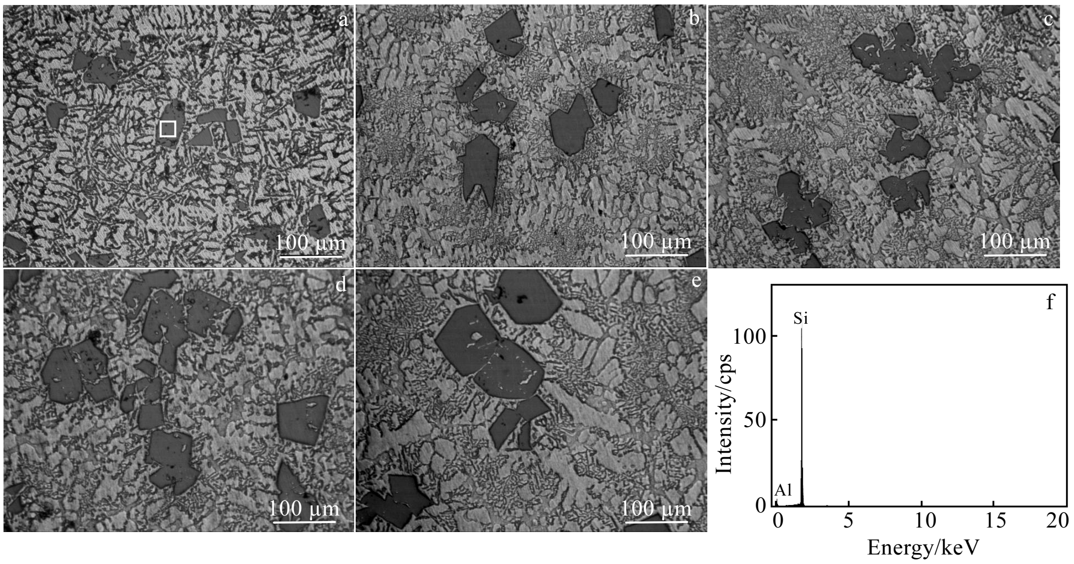

Fig.3 Microstructures of primary silicon phase with different Sr and Ca additions: (a) sample A, (b) sample B, (c) sample C, (d) sample D, (e) sample E, and (f) EDS spectrum corresponding to rectangle area in Fig.3a

alloy. In the unmodified A390 aluminum alloy, as shown in Fig.3a, primary silicon phase mainly presents as polygon lump, such as quadrilateral, pentagon and/or hexagon. This kind of morphology accords with the TPRE growth mechanism of silicon crystal[20], i.e., the primary silicon phase is of octahedron growth on {111} plane in near-equilibrium solidification condition, and the two-dimensional section is polygonal. When A390 aluminum alloy was modified with 0.05% Sr, as shown in Fig.3b, the size of primary silicon phase increases slightly, and the shape remains polygonal with distinct edges. As evident from Fig.3c, the primary silicon phase exhibits fragmentation, and the primary silicon phase size is refined, which contributes to reducing the brittleness of the primary silicon phase when Ca is used to partly substitute for Sr. However, as shown in Fig.3d~3f, the primary silicon phase size increases obviously and the segregation phenomenon occurs with the increase in Ca addition.

The modification effects of Sr-Ca complex on the primary silicon phase in A390 aluminum alloy are quantified by the factors of volume fraction (φ), equivalent diameter (D_P) and shape factor (S_F), as shown in Eq.(1)~(3), which were obtained by analyzing the images using Image-Pro Plus 6.0 image analyzer. (D_P) is the equivalent circle diameter of the particle area, and it is not sensitive to the particle shapes. As a result, it can accurately characterize the size of particles. The value of the shape factor S_F is between [0, 1]. If the value is close to 1, the shape of the particle is more similar to the ideal sphere. These factors may be represented by the following equations:

$$\varphi = \frac{A_T}{A_0} \tag{1}$$

$$D_P = \sqrt{\frac{4A_P}{\pi}} \tag{2}$$

$$S_F = \frac{4\pi A_P}{C^2} \tag{3}$$

Where A_P is the area of a single primary silicon phase, A_T is the sum area of all primary in the field of view, A_0 is the total area of the rectangular field of view, C is the perimeter of the primary silicon phase.

Fig.4 depicts quantitative metallographic results of primary silicon phase in A390 aluminum alloy. With combined addition of 0.05%Sr-Ca, φ and S_F firstly increase and then decrease, while D_P shows the trend of decrease first and then increase on the contrary. The three curves reach the extreme point when the atomic ratio of Sr and Ca is 5:1. After addition of 0.05% Sr, the φ and S_F decrease from 9.10% and 0.80 (unmodified alloy) to 6.91% and 0.77, while the D_P increases from 48.31 to 54.11 μm, respectively, which indicates that the primary silicon phase quantity decreases, the size enlarges and the morphology grows irregularly, resulting from the addition of Sr. Contrast samples B and C, Sr and Ca content of sample C is higher than that of sample B and the indicators of primary silicon are more excellent, namely the primary silicon modification effect is better in the sample C. Sr has an adverse effect on the modification of primary silicon, because the TPRE growth mechanism of primary silicon is inhibited when Sr is added[21], so it can be inferred that Ca plays a major role in the modification of primary silicon. When the amount of Ca is 0.0025%, the optimal morphology of primary silicon phase forms and φ, D_P and S_F are 14%, 49.54 μm and 0.83, respectively. When the Ca content exceeds 0.0079%, the D_P is as high as 64.35 μm. These results suggest that Ca addition can compensate for the increased size of primary silicon phase; however excessive Ca content results in modification deficiency owing to the formation of new stable product $CaSi_2$ with the melting point 1020 °C, which reduces the effective content of modifier in the alloy[22].

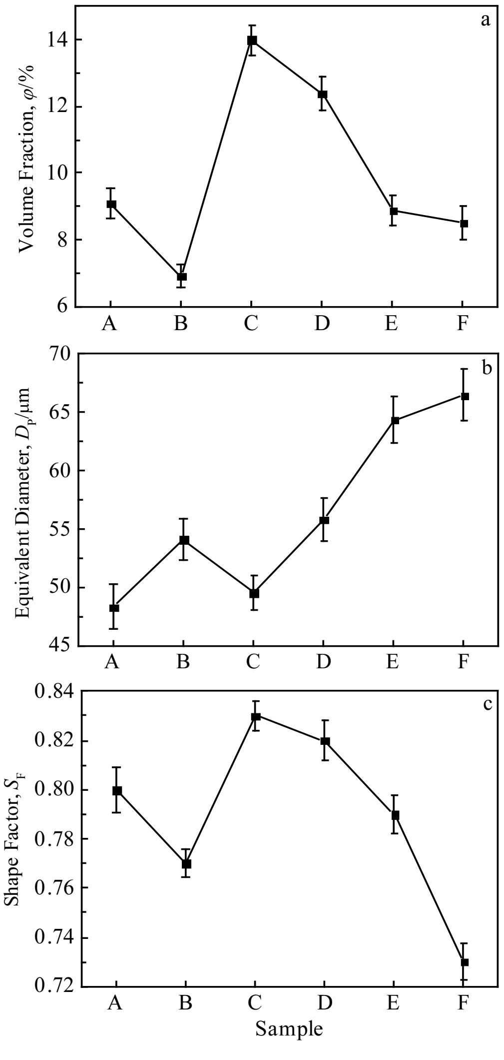

Fig.4 Impacts of primary silicon phase after Sr and Ca addition: (a) φ, (b) D_P, and (c) S_F

The above analysis shows that Sr will increase the size of primary silicon phase, while the addition of a small amount of Ca inhibits this coarsening effect. With the increase in Ca content, the D_P of primary silicon phase rises and the φ decreases, i.e., the number of primary silicon phase decreases gradually in unit area. It is indicated that the addition of Ca has no effect on nucleation. Accordingly, Ca is unlikely to be the core of primary silicon phase. The study of Lu and Hellawell[23] have shown that the closer the modifier radius to the silicon atom radius, the better the modification is. According to the impurity induced twinning theory[24], the radius ratio of Ca atom and Si atom is 1.68; therefore, Ca has definite possibility to play a role in modification through adsorbing to the growth interface of silicon phase, which inhibits the growth mechanism of the twin groove of primary silicon phase, and induces new twins at the same time.

2.3 Effect of Sr-Ca combined addition on eutectic silicon phase

The morphologies of eutectic silicon phase with different Sr and Ca addition in A390 aluminum alloy are shown in Fig.5. Coarse needle-like eutectic silicon phase, about 20~50 μm in length and nearly 1.5~6.0 μm in width, with the characteristics of small plane growth, can be clearly observed in the unmodified A390 aluminum alloy, as shown in Fig.5a. It can be seen in 0.05% Sr addition results in significant refinement of the microstructure, where most of the eutectic silicon phase changes into fibrous, yet part of them keeps needle-like as usual (shown in the arrow in Fig.5b). It is worthwhile mentioning that the eutectic silicon phase exhibits aggregation because the silicon phase is no longer the leading phase, the growth front of the silicon phase and α phase is almost an isothermal flat interface and the boundaries of eutectic cluster are formed by intermetallic compounds with Sr addition. With combined addition of 0.05%Sr-Ca and the atomic ratio of 5:1, as shown in Fig.5c, the modification effect is improved obviously as the needle-like parts disappears and all of them change into fibrous eutectic clusters. But when reducing the Sr and Ca atomic ratio, i.e. increasing the amount of Ca addition, the coarse needle-like eutectic silicon phase appears again, the size of eutectic clusters increases and the modification effect deteriorates, as shown in Fig.5d. Thereby, under the premise that total amount of the modifier remains constant, the change of Sr and Ca ratio results in variable modification effects, which can be clearly demonstrated by quantitative metallographic analysis.

The morphology of eutectic silicon phase was characterized by the slice spacing (λ) and the average of maximum length (L), and the size of eutectic silicon phase was characterized eutectic cluster by equivalent diameter (D_E), as shown in Eq.(4).

$$D_E = \sqrt{\frac{4A_E}{\pi}} \tag{4}$$

Where A_E is the area enclosed by the eutectic cluster boundary.

The quantitative influence of Sr-Ca composite modification on D_E, λ and L of the eutectic silicon phase is shown in Fig.6. The results reflect that D_E, λ and L of the eutectic silicon phase in A390 aluminum alloy present a trend of decreasing first and then increasing. The eutectic silicon phase was significantly improved after 0.05%Sr addition compared with before; D_E, λ and L of the eutectic silicon phase change from 173.41, 4.71 μm and 35.01 to 114.64, 3.27 and 13.84 μm, respectively. When Sr and Ca atom ratio is 5:1, the optimal morphology of the eutectic silicon phase is

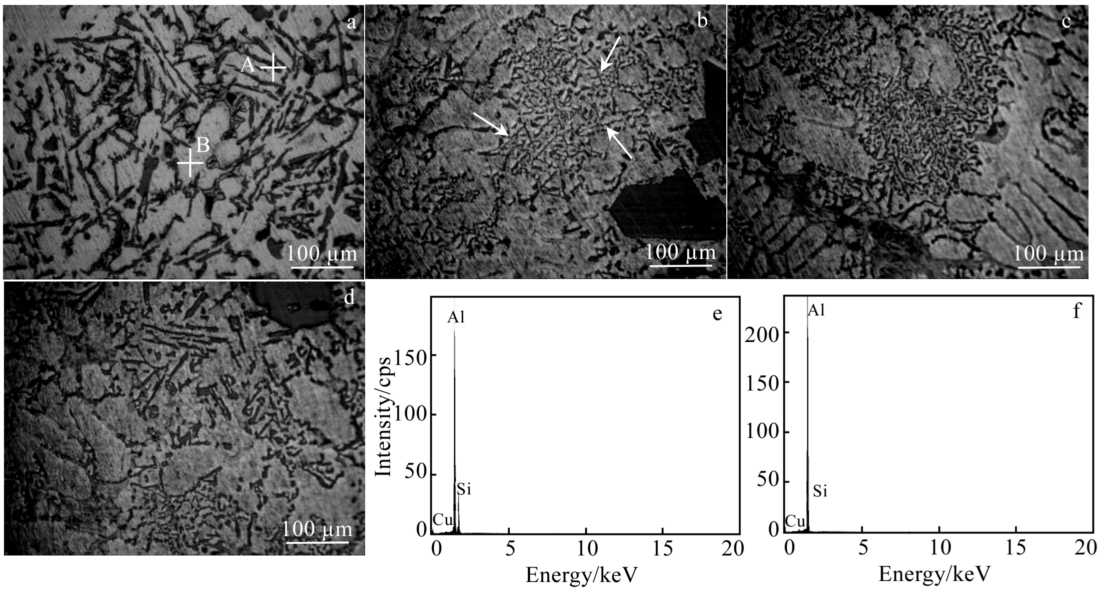

Fig.5 Microstructures of eutectic silicon phase with different Sr and Ca additions: (a) sample A; (b) sample B; (c) sample C; (d) sample D; EDS spectra corresponding to cross point A (e) and point B (f) in Fig.5a

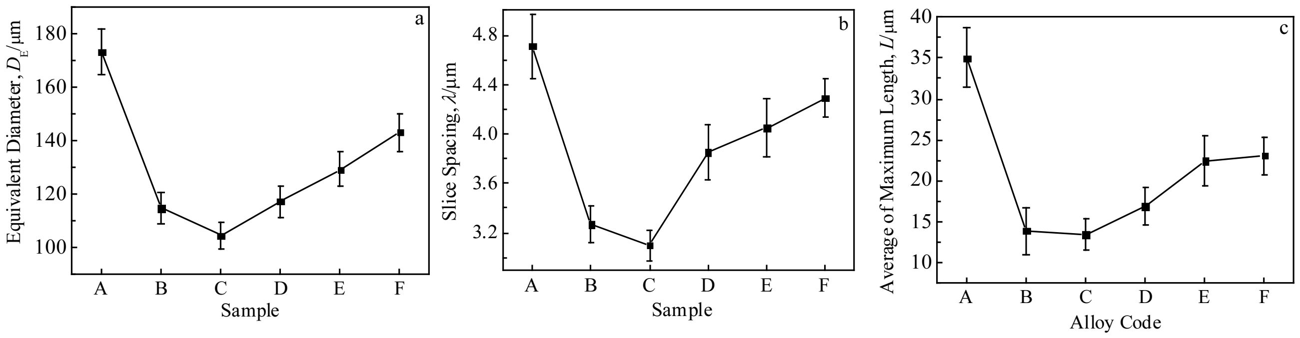

Fig.6 Impacts of eutectic silicon phase after Sr and Ca addition: (a) D_E, (b) λ, and (c) L

obtained and D_E, λ and L of the eutectic silicon phase are 104.39, 3.10 and 13.37 μm, respectively, given that the addition of Ca promotes the effective absorption of Sr and increases the Sr content in the melt, and thus the modification of Sr is enhanced. When the Sr and Ca atom ratio decreases further, i.e., the content of Ca in the melt increases and the content of Sr decreases, D_E, λ and L of the eutectic silicon phase are gradually increased. However, since the Sr is always present in the melt, the eutectic silicon phase is superior to that of the unmodified form.

Experimental studies have demonstrated that the D_E, λ and L of the eutectic silicon phase all exhibit the trend of decreasing first and then increasing, which is consistent with the change of Sr content in the melt. There is no denying the fact that Sr has a modification effect on the eutectic silicon phase. The Al_2Si_2Sr phase formed by Sr addition in the melt is enriched at the front of the silicon phase, which changes the growth pattern of eutectic silicon phase and transforms it from plate-like to fibrous[4]. Furthermore, Sr can increase undercooling, i.e., increasing the driving force of crystal growth; thereby the solidification rate increases and the size of eutectic clusters[25]. The effect of Sr-Ca complex modification on the eutectic silicon phase is mainly due to the addition of Ca, which increases the content of Sr in the melt within the optimum range, and thus the modification effect of Sr is facilitated.

2.4 Effect of Sr-Ca combined addition on primary α phase

The size and morphology of the primary α phase were characterized by the second dendrite arm spacing λ_2. Measurements of primary dendrite spacing were carried out on the cross section, and measurements were generally made at 5 mm below the solid-liquid interface. A large enough rectangular area was taken on the intersecting surface, whose boundary was defined in the middle of the dendrite.

The measurement of the second dendrite arm spacing was calculated on the vertical section with uniform arrangement and consistent with orientation of dendrites, which allows the second dendrite attaching to the same dendrite. The second dendrite arm spacing is expressed by λ_2.

Fig.7 shows the effect of Sr-Ca complex modification on the primary α phase in A390 aluminum alloy. The curves of λ_2 reflects the trend of decreasing first and then increasing. When only 0.05% Sr is added, λ_2 is reduced by 11.52% compared to the unmodified A390 aluminum alloy, i.e., the primary α dendrites become finer and the distribution is more compact. When combined addition of 0.05%Sr-Ca and the atomic ratio is 5:1, the minimum λ_2 is obtained at 14.40 μm. When the Sr and Ca atomic ratio decreases further, λ_2 increases significantly. The addition of Ca reduces the secondary dendrite arm spacing, which mainly determines the properties of alloy; therefore, the trace Ca addition is advantageous to the primary α phase morphology. Based on the diffusion kinetics under normal solidification conditions, Ca elements will gather in front of solid liquid interface, cause solute redistribution and increase the alloy solidification undercooling during the solidification process, which intensifies the branching process and reduces the secondary dendrite arm spacing[26].

2.5 Effect of Sr-Ca combined addition on intermetallic phase

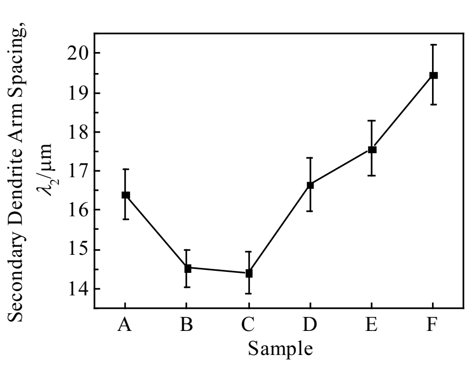

Fig.7 Secondary dendrite arm spacing λ_2 of α-Al phase after Sr and Ca addition

Fig.8 shows the morphology and the distribution of the intermetallic phases. A390 aluminum alloy with different additions of Sr and Ca. The intermetallic phase is identified as θ phase and β-Fe phase which are distributed between the primary α phase dendrites as these were identified in the previous paper[25]. The θ phase exhibits coarse coral and the β-Fe phase is slender needle-like in the A390 aluminium alloy, as shown in Fig.8a. The θ phase is refined remarkably and the β-Fe phase remains slender needle-like with 0.05% Sr addition, as shown in Fig.8b. With the Sr and Ca atomic ratio decreasing, the θ phase gets coarser and coarser, while the β-Fe phase is unchanged with 0.05% Sr and Ca combined addition, as evidenced from Fig.8c and 8d. The results imply that Sr has a refinement effect on the θ phase. The combination of Sr and Ca makes the θ phase coarsen and has no effect on the β-Fe phase. Further experimental study is needed on the principle of the coarsening of the θ phase.

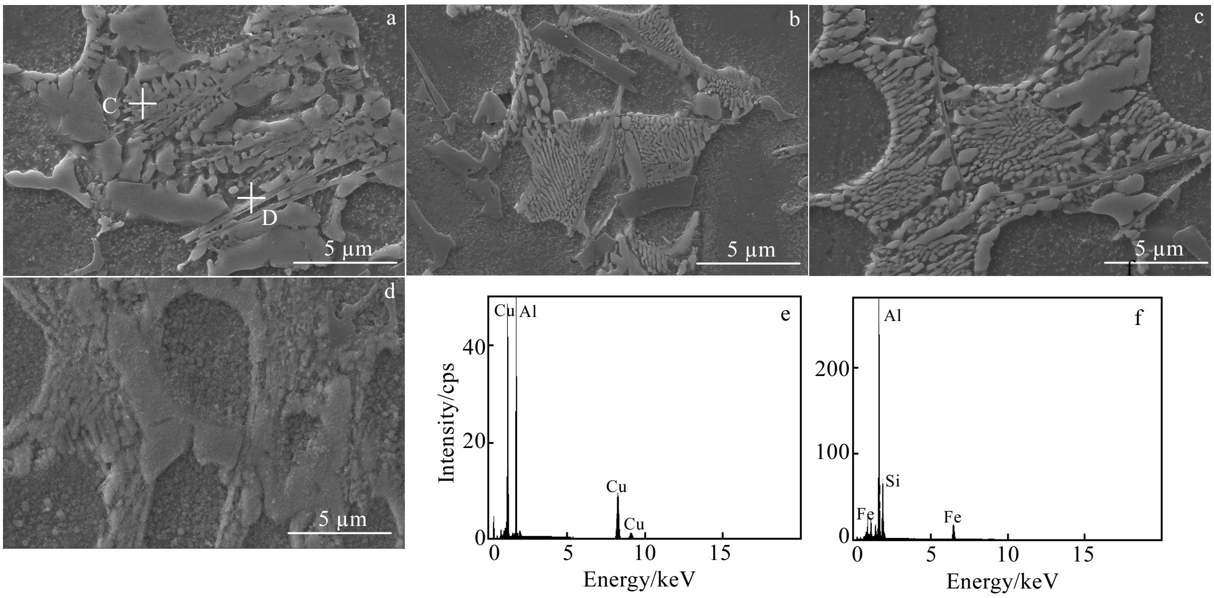

Fig.8 Microstructures of θ phase and β-Fe phase after Sr and Ca addition: (a) A390; (b) 0.05%Sr; (c) 0.05%Sr-Ca, $n_{\mathrm{Sr}}:n_{\mathrm{Ca}}=5:1$; (d) 0.05%Sr-Ca, $n_{\mathrm{Sr}}:n_{\mathrm{Ca}}=2.5:1$; EDS spectra corresponding to cross point C (e) and point D (f) in Fig.8a

3 Conclusions

1) The synergistic effect on the microstructure of A390 aluminum alloy results from complex modification of Sr-Ca. The Sr loss rate is slowed down with a small amount of Ca; meanwhile, coarsening effect of primary silicon phase arising from Sr addition is suppressed. The synergistic effect is most prominent when the atomic ratio of Sr and Ca is 5:1.

2) The effect of Sr in A390 aluminum alloy is mainly modifying eutectic silicon phase and refining θ phase, so that the eutectic silicon phase changes from plate-like to fibrous, and agglomerate phenomenon occurs when Sr and Ca atom ratio is comparatively low.

3) Ca has a favorable modifying effect on the primary silicon phase and on the primary α phase in A390 aluminum alloy. A small amount of Ca addition can reduce the size of primary silicon phase and decrease the secondary dendrite arm spacing of primary α phase. The best modification effect is attained when the content of Ca is 0.0025%, and the phenomenon of excessive modifying will occur when the content of Ca exceeds 0.0079%.

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Sr-Ca 复合变质对 A390 铝合金微观组织的影响

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摘　要：研究了 Sr-Ca 复合变质对 A390 铝合金显微组织中初生硅相、共晶硅相、初生 α 相和金属间化合物相数量、尺寸和形貌变化的影响规律。结果表明，Sr-Ca 复合变质体现为协同作用，少量 Ca 的加入不仅减缓了 Sr 在熔体中的烧损，降低了 Sr 烧损率，而且抑制了 Sr 对初生硅相的粗化作用。在 A390 铝合金中 Sr 的主要作用是变质共晶硅相和细化 θ 相，Ca 的主要作用是变质初生硅相和初生 α 相。当联合添加质量分数 0.05%Sr-Ca 且 Sr、Ca 原子比为 5:1 时，获得了最佳组织形态，其中初生硅相、共晶硅相、初生 α 相均较 0.05%Sr 单独变质时有明显改善。

关键词：Al-Si合金；变质；晶粒细化；微观组织

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