

Effect of Casting Parameters on Microstructure and Casting Quality of Si-Al Alloy for Vacuum Sputtering

IN-TING HONG, HUAN-CHIEN TUNG, CHUN-HAO CHIU and HUNG-SHANG HUANG

*Steel and Aluminum Research and Development Department
China Steel Corporation*

This investigation studies the effect of casting parameters on the casting quality of Si-30wt.% Al alloy used for vacuum sputtering. The practical casting results were compared with simulation results analyzed by Flow-3D software to clarify the forming reason of casting defects and to further improve the casting process. Moreover, the Si-Al alloys with different Al contents were sputtered on a glass substrate to form Si-Al thin films to investigate the effect of Al content on the Non-Conductive Vacuum Metallization (NCVM) property of Si-Al thin film. The simulation results analyzed by Flow-3D software indicate that a large amount of shrinkage pores form in the ingot, as the Si-30wt.%Al liquid is being cast into the thick mold. The shrinkage pores in Si-Al ingot can be greatly reduced by employing a thin mold. However, some casting pits can be observed on the surface of ingot which was cast into the thin mold, because the liquid is hard to precisely cast into the cavity of the thin mold. The simulation results of Flow-3D software reveal that a "hot spot" appears near the riser of the thin mold, as the liquid is cast on the riser and then flows into the cavity of thin mold. Some casting pits exist near the hot spot due to the liquid temperature near the hot spot being too high. In addition, a U-shaped solidification defect which can be completely eliminated by employing a tundish to steady casting flow exists on the surface of ingot, as the casting flow suddenly increases during casting.

1. INTRODUCTION

Plastic materials, which are light, easy to mass produce, shape-flexible and do not influence signal transmission, were widely used for 3C product casings. However, plastic materials do not show the shine of metal and are not eye-catching to customers. Non-Conductive Vacuum Metallization (NCVM) sputtering technology can equip plastic materials with a metallic shininess and mirror effect and improve the visual quality of plastic material, thereby becoming the main process to improve the appearance and quality of plastic casings used for 3C products.^(1,2)

Si-Al alloy is an ideal material for the NCVM process because the thin film sputtered by Si-Al alloy not only possesses a metallic shininess but also do not affect the signal transmissions of 3C products. The addition of Al to Si gives an Si-Al thin film a metallic shine. However, Al addition not only increases the casting difficulty of an Si-Al alloy, but also decreases the electrical resistance of an Si-Al thin film, and its signal transmission effect.

Serious segregation and a large amount of shrinkage pores always exist in the Si-Al ingot after casting due to the large difference between the solidus and liquidus temperature of Si-Al alloy. Segregation and shrinkage pores existing in the target cause arcing problems during sputtering and, moreover, influence the properties of the Si-Al thin film. To clarify the effect of casting parameters on the casting quality of Si-Al alloys, practical casting results were compared with simulation results analyzed by Flow-3D software. The parameters include mold designation, casting temperature, mold temperature and casting location. Moreover, Si-Al alloys with different Al contents were sputtered on the glass substrate to observe the morphology of the Si-Al thin film, and to investigate the effect of Al content on the NCVM property of the Si-Al thin film.

2. EXPERIMENTAL METHOD

To understand the effect of Al content on the microstructure of Si-Al alloy and the properties of Si-Al thin film, Si-Al alloys containing 20, 25, 30 and 35 wt.% Al were melted by vacuum induction melter and

then cast into the rectangular thin molds. After cooling, the Si-Al ingots were manufactured into circular sputtering targets with 6 inch diameter. The Si-Al targets were sputtered to form Si-Al thin films with different Al contents on the glass substrate. The n&k analyzer 1280 was used to measure the reflection rate of the Si-Al thin films within the wave length range of 200 to 1000nm to obtain the optimum composition and thickness of the Si-Al thin film with the best reflection rate. Moreover, the Si-30wt.%Al target was sputtered on the glass substrate with different working power. The atomic force microscopy (AFM) and scanning electron microscopy (SEM) were used to observe the morphology of Si-Al thin film to determine the effect of working power on the morphology of the Si-Al thin film. The microstructure of the Si-Al targets was observed by optical microscopy (OM). The percentage of shrinkage pores of the Si-Al target was calculated by image analysis software.

Serious segregation and a large amount of shrinkage pores always appear in the Si-Al ingot because of the large difference between the solidus and liquidus temperatures of the Si-Al alloy. Therefore, a thin mold should be used to cast Si-Al alloy to speed up the solidification rate of the Si-Al liquid. To analyze the reasons for the formation of casting defect via CAE software, the 3D schema of casting molds were plotted by Solidworks software, as shown in Figure 1. The Flow-3D software was used to simulate the casting process and to clarify the effects of the casting parameters on the casting quality of the Si-Al target. These simulation results were used to improve the practical casting process.

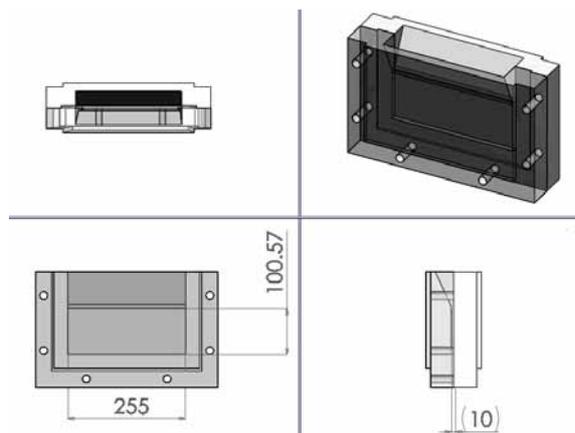


Fig. 1. Schema of casting mold plotted by Solidworks software. (unit:mm)

3. RESULTS AND DISCUSSION

3.1 Effect of Al Content on Properties of Si-Al thin Film

NCVM can equip plastic materials with a metallic

sheen. Meanwhile, the thin film sputtered by the NCVM process does not affect signal transmission due to its high resistance, so that NCVM process is becoming a mainstream sputtering technology for improving the appearance of plastic materials^(1, 2). Si-Al thin films not only possess a high reflection rate but also high electric resistance, thus Si-Al alloys are widely used as sputtering target for NCVM process.

Figure 2 shows the reflection rate of Si-Al thin films with different Al contents within the wave length range from 200 to 1,000nm. When the Al content is increased from 20 to 30wt.%, the reflection rate of Si-Al thin film within the wave length of visible light increases in line with Al content, implying that the high reflection rate of the Si-Al thin film results from the Al addition. Although Al addition does increase the conductivity of Si-Al thin film, the resistance of the Si-Al thin films containing 20 to 30wt.%Al are all above 1 mega-Ohm. However, as the Al addition further increases to 35wt.%, the resistance of the Si-Al thin film decreases significantly to about 0.7 mega-Ohm, which is lower than the standard for the NCVM process. To make the resistance of the Si-35wt.%Al thin film higher than 1 mega-Ohm, to meet the standard of the NCVM process, the thickness of Si-Al thin film must be reduced to 15nm. Compared with the other three Si-Al thin films whose thicknesses are all around 20nm, the reflection rate of Si-35wt.%Al thin film is the lowest due to its thickness being just 15nm to make it insulated. Therefore, the Al content of Si-Al thin film should be controlled around 30wt.% to equip an Si-Al thin film with a high reflection rate and acceptable insulating property.

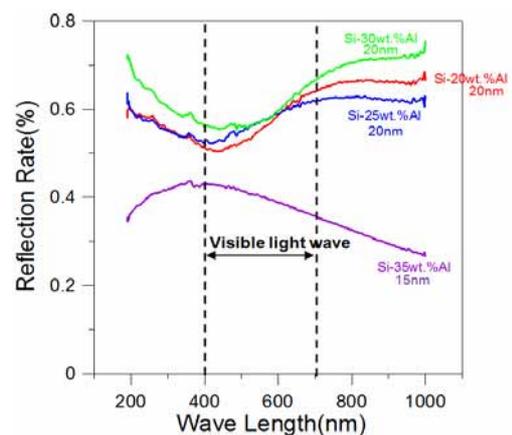


Fig. 2. Reflection rate of Si-Al thin films with different Al contents.

Figure 3 shows the SEM and AFM images of Si-30wt.%Al thin film sputtered by different working powers. The Si-30wt.%Al thin film agglomerates when the Si-Al target was sputtered with a 1,000W working power, as shown in Figure 3(a). The morphology of the Si-30wt.%Al thin film becomes smoother as the

working power decreases. The Si-30wt.%Al thin film sputtered with 300W working power is the flattest, as shown in Fig. 3(c). Generally, flat thin films can reflect light more effectively, so flatter thin films possess higher reflection rates, showing more metallic shininess. For Si-30wt.%Al thin film, a better metallization effect can be obtained by sputtering Si-Al target with a lower working power.

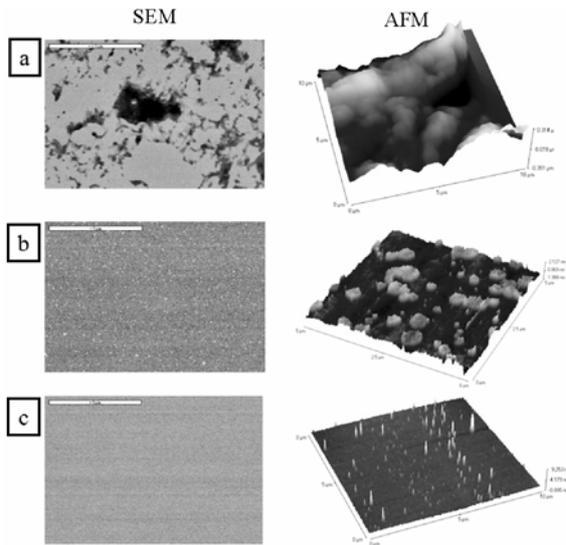


Fig. 3. SEM and AFM images of Si-Al thin film sputtered by Si-30wt.%Al target with different working power: (a) 1,000W; (b) 500W; and (c) 300W.

3.2 Effect of Al Contents on Microstructure of Si-Al thin Film

Figure 4 shows the microstructure of Si-Al alloy with different Al contents. The results reveal that shrinkage pores tend to form in the pre-eutectic Si phase during solidification, and the amount of shrinkage pores increases with the Al addition. The casting characteristics of Si-Al alloy are quite different from those of Al-Si alloy. The Al-Si alloys containing 2 to 13

wt.% Si solidify sequentially because the difference between the solidus and liquidus temperatures of Al-Si alloy is just about 50 to 90 . During solidification, shrinkage pores do not exist throughout the ingot because the liquid can flow into and fill the space between the solid phase. The shrinkage pores can be effectively avoided in these Al-Si alloys by a well-designed riser. However, the difference between the solidus and liquidus temperatures of Si-25 to 35 wt.%Al is as high as 650 to 700 , as shown in Fig. 5(a). For Si-Al alloy containing 25 to 35 wt.%Al, the pre-eutectic Si phase solidifies first and connects with each other during solidification. The liquid phase is cut into several separated small pools by the pre-eutectic Si phase. During solidification, these small pools solidify and form shrinkage pores because no liquid can flow into, and fill, these isolated pools, as shown in Fig. 4.

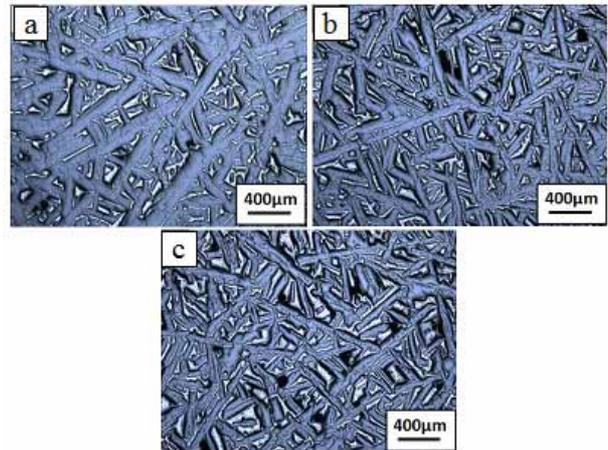


Fig. 4. The OM image of Si-Al targets containing different Al contents: (a) 25wt.%; (b) 30wt.%; (c) 35wt.%

Si and Al do not dissolve with each other, and the solidification partition coefficient of Si-Al alloy is smaller than 1, so that during solidification the liquid firstly solidifies to form pre-eutectic Si, ejecting the Al

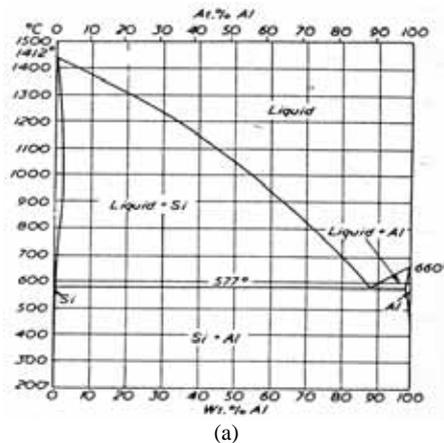


Fig. 5. (a) Si-Al phase diagram; (b) The macro-segregation of Si-Al ingot.

element into the un-solidified liquid. Meanwhile, the un-solidified liquid is pushed by the solidified pre-eutectic Si and moves upward towards the top surface of ingot. Finally, the aluminum-rich liquid emerges from the riser of the ingot and forms the macrosegregation, as shown in Figure 5(b). The composition of the bulk analyzed by Wavelength-dispersive electron probe microanalysis (WDS-EPMA) is about Si-84.7wt.%Al, close to the composition of eutectic in Si-Al phase diagram.

3.3 Effect of Mold Designation and Tundish on Casting Quality of Si-30wt.%Al Target

During solidification, most alloys possess different phases, including solid phase, solid-liquid phase (mush zone), and liquid phase, except for pure metals and eutectic alloys⁽¹⁻³⁾. For alloys with a great difference between the solidus and liquidus temperatures, the solid phases which solidify first connect with each other, preventing the liquid from flowing into the spaces between the solid phases.

During solidification, the width of the mush zone of alloy is proportional to $\frac{T_l - T_s}{G}$ ⁽⁴⁾, where T_l and T_s are the liquidus and solidus temperatures, and G is the temperature gradient of the liquid in the mold after casting.

The width of mush zone of Si-Al alloy is quite large because Si-Al alloy possesses a large temperature gap between solidus and liquidus, so that a large amount of shrinkage pores exist in the ingot after solidification. As the width of the mush zone reduces, the liquid flows more easily across the mush zone, so that the amount of shrinkage pores in Si-Al alloy can decrease. Therefore, the amount of shrinkage pores in Si-Al alloy can be reduced by increasing the temperature gradient (G). The temperature gradient (G) can be expressed as follows:

$$G = \frac{-Rm_L(C_L^* - C_S^*)}{D_L}$$

Where R is the solidification rate of liquid, m_L is the slope of liquidus line of phase diagram, C_S and C_L are the composition of the solid and liquid at the interface, and D_L is the diffusion coefficient of solute in the liquid. Therefore, as the cooling ability of the mold improves, the temperature gradient G is increased with the solidification rate of liquid, and thus the width of mush zone reduces. Once the width of mush zone reduces, the liquid flows more easily to fill any spaces, resulting in a significant decrease of shrinkage pores.

Figure 6 is the simulation results analyzed by Flow-3D of casting Si-30wt.%Al into a thick mold. The simulation results reveal that a large amount of

shrinkage pores exist in the middle of ingot, as the Si-30wt.%Al is cast into the thick mold. This result implies that the solidification rate of the liquid in the middle of ingot is too slow to avoid the formation of shrinkage pores. Therefore, to reduce the shrinkage pores, a thin mold should be used to cast Si-Al alloy to speed up the solidification rate of the liquid.

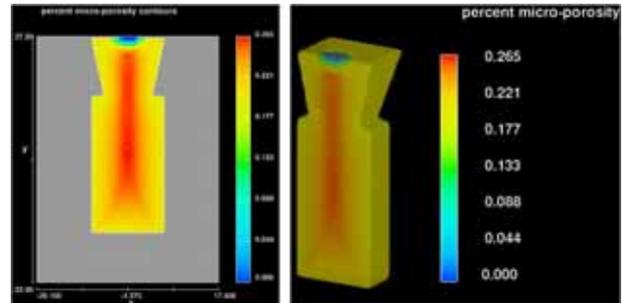


Fig. 6. Results of Flow-3D simulation of Si-Al alloy cast into a thick mold.

However, when a thin mold was used for casting, the liquid could not be cast into the mold cavity precisely. The liquid was always cast onto the riser and then flowed into the mold cavity, because the mold cavity is too thin to cast accurately. The practical casting results show that the casting location affects the casting quality of Si-Al alloy significantly. No shrinkage pores appear on the surface of the ingot when the liquid was cast into the mold cavity directly. However, a large amount of shrinkage pores appear on the surface of the ingot if the liquid is cast onto the riser and then flowed into the mold cavity.

Figure 7 is the simulation result of casting Si-30wt.%Al directly into the mold cavity, showing that two areas with lower temperature exist in the liquid at the beginning of casting. The distribution of liquid temperature is relatively uniform due to heat circulation when the liquid fills up the whole mold. However, if the liquid is cast onto the riser and then flows into the mold along the mold wall, a “hot spot” appears around the riser because the high-temperature liquid continually contacts the mold wall. When the liquid has filled up the whole mold, the temperature of the liquid around the “hot spot” is about 300 higher than that of other areas, as shown in Fig. 8.

During solidification, most casting defects existing in the ingot are closely related to the temperature of liquid. If the liquid temperature is too high, the shrinkage of the ingot is increased, and shrinkage pores form in the ingot.⁽⁵⁻⁷⁾ Thus, liquid with a higher temperature always takes longer time to solidify, forming more shrinkage pores.⁽⁷⁾

The temperature of the liquid around the “hot spot” caused by imprecise casting is higher than that in

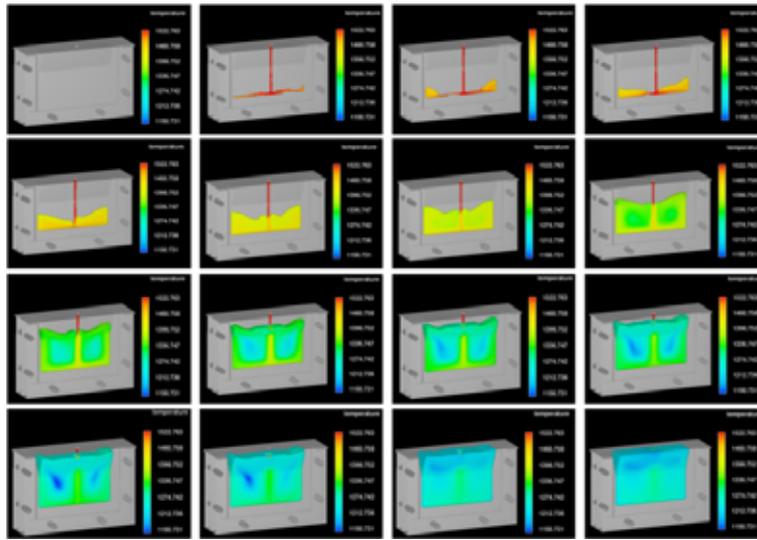


Fig. 7. Temperature distribution of melt cast into the cavity of mold directly.

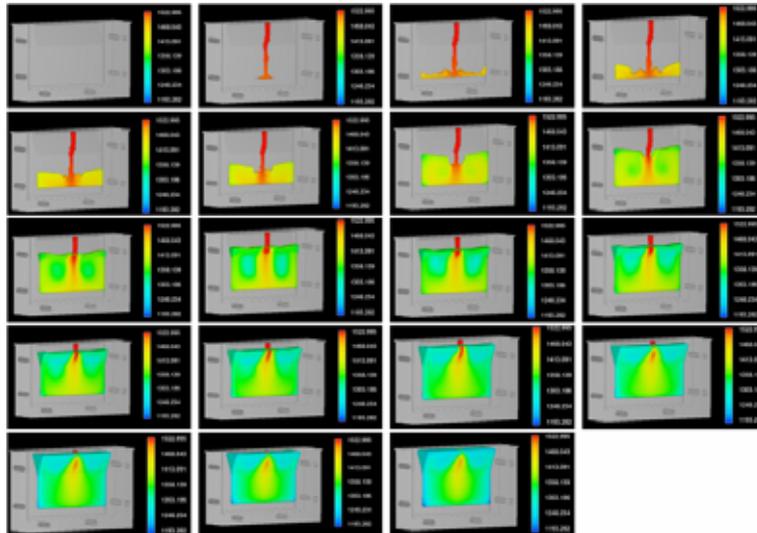


Fig. 8. Temperature distribution of melt cast onto the riser with subsequent flow into the mold cavity.

other areas. After solidification, a large amount of shrinkage pores exist around the “hot spot” because the liquid temperature in this area is too high. To ensure that the high temperature liquid flows directly into the mold with minimal contact of the mold wall, a tundish was used for casting Si-Al alloy. The tundish, as shown in Figure 10(a), can stabilize the liquid flow and control the casting location, and thus avoid the shrinkage pores caused by imprecise casting. No shrinkage pores appeared on the surface of the ingot cast with a tundish, as shown in Fig. 10(b).

3.4 Effect of Casting Parameters on Casting Defects of Si-30wt.% Al Target

The optimum casting temperature of an alloy depends on its composition and solidification character. The casting temperature is always set as 40 to 150 higher

than the melting point of the alloy.⁽¹⁻³⁾ The casting temperature directly affects the quality of casting ingot. A higher casting temperature can increase the fluidity of liquid, making the liquid fill up the mold smoothly. However, a higher casting temperature not only leads to coarse grain, but also increases the solidification shrinkage and amount of shrinkage pores in an ingot. Mold temperature is another key factor which affects the casting quality of an alloy.⁽⁴⁻⁵⁾ In order to avoid the cold shut and gas pores, a mold needs to be pre-heated to keep the mold at a certain temperature before casting. The setting of the mold temperature depends on solidification character of the alloy. For an alloy which possesses a higher melting temperature or worse fluidity, the mold temperature should be moderately increased. In contrast, for an alloy which tends to form segregation or possesses a large difference between the solidus and

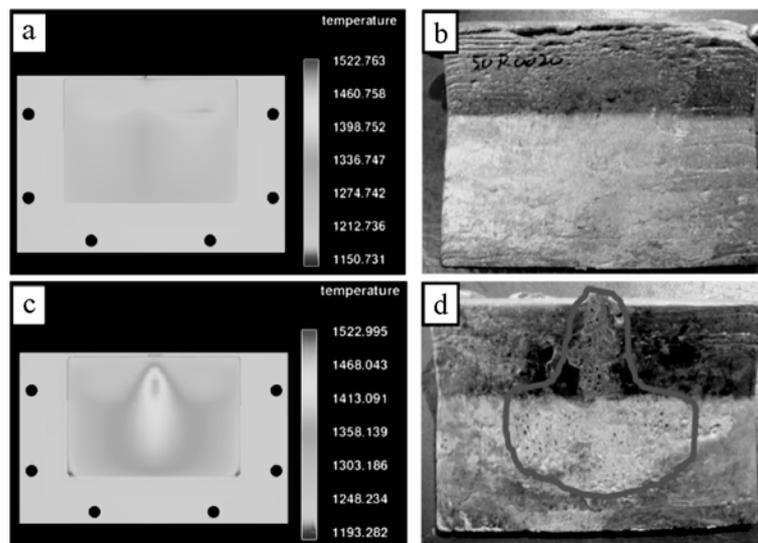


Fig. 9. Relation between the temperature distribution of melt and shrinkage pores on the surface of an ingot.

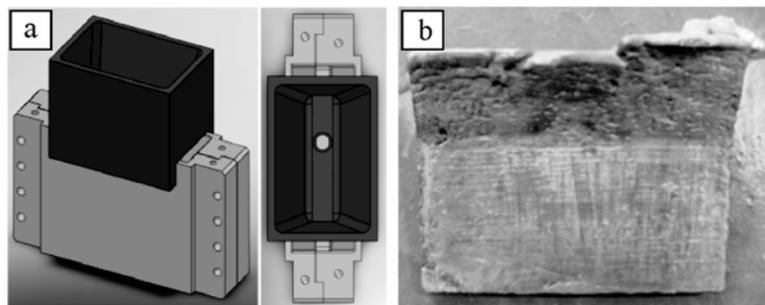


Fig. 10. (a) Schema of tundish; (b) Surface of Si-30wt.%Al ingot cast with tundish.

liquidus temperatures, the mold temperature should be moderately decreased to avoid shrinkage pores and segregation⁽⁵⁾.

Figure 11 shows the casting results of Si-30wt.%Al cast at 1,330 °C with a mold temperature for 300 °C, revealing that a large amount of Al segregation exists on the surface of the ingot, as shown in Fig.11(a). A large number of shrinkage pores exist in the center of ingot, and the percentage of shrinkage pores is as high as 16%. The above results can be attributed to the casting and mold temperatures being too high. Higher casting and mold temperatures increase the shrinkage of liquid and decrease the solidification rate of liquid, respectively, so that Al segregates on the surface of the ingot. When the casting and mold temperatures are decrease to 1,330 °C and 250 °C, respectively, the Al segregation still appears on the surface of the ingot, but the percentage of shrinkage pores decreases to 11%, as shown in Fig. 12(a) and 12(c). If the mold temperature is further decreased to 150 °C and the casting temperature still kept at 1,300 °C, the Al segregation on the surface of the ingot is significantly reduced, and the percentage of shrinkage pores decreases to 9%. Further, as the casting temperature and mold temperature are decreased to 1,270 °C and

50 °C, respectively, no Al segregation can be observed on the surface of the ingot and the percentage of shrinkage pores decreases to 4%, as shown in Fig. 14. The above results reveal that the casting temperature and the mold temperature are the key factors to casting quality of Si-Al alloy. The Si-Al phase diagram shows that the melting point of Si-30wt.%Al is 1,230 °C. When the

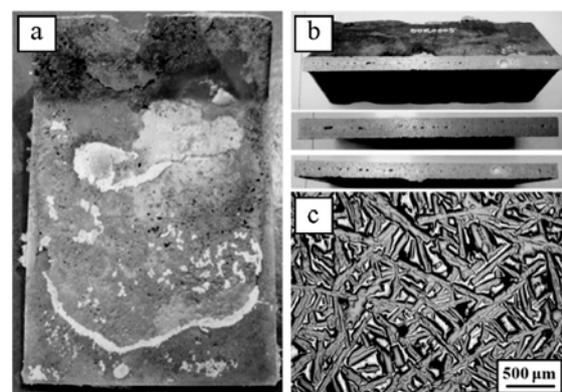


Fig. 11. Shrinkage pores on the (a) surface; and (b) center of Si-30wt.%Al ingot cast with the condition of 1330 °C casting temperature and 300 °C mold temperature. (c) OM observation and the distribution of shrinkage pores.

casting temperature and the mold temperature are higher than 1,300 and 200 , respectively, a large amount of shrinkage pores will exist in the ingot, and Al segregation can be observed throughout the ingot.

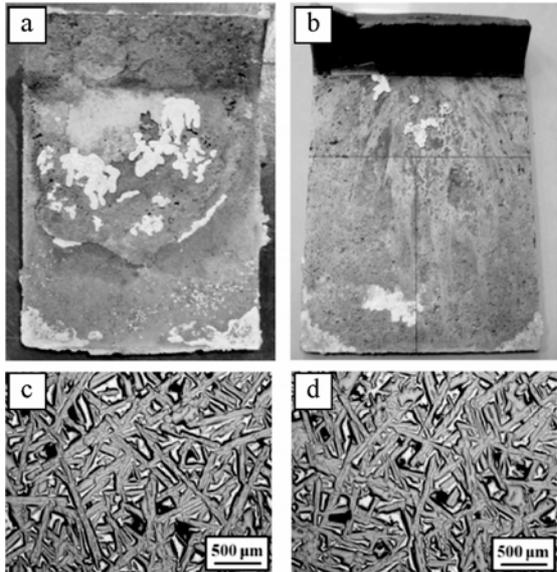


Fig. 12. Surface condition of the Si-30wt.%Al ingot cast with the condition of 1300 casting temperature and (a) 250 mold temperature, (b) 150 mold temperature. (c),(d) OM observation and the distribution of shrinkage pores at 250 and 150 mold temperature, respectively.

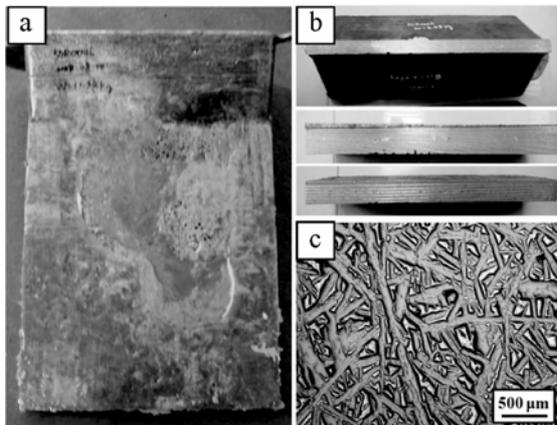


Fig. 13. Shrinkage pores on the (a) surface and (b) center of Si-30wt.%Al ingot cast with the condition of 1270 casting temperature and 50 mold temperature. (c) OM observation and the distribution of shrinkage pores.

Moreover, a U-shaped casting defect appears on the surface of ingot if the casting flow is suddenly increased during casting. Al segregation and shrinkage pores can be observed around the U-shaped casting defect.

Figure 14 shows the simulation results of the turbulent energy of a liquid that was cast into the mold smoothly. The turbulent energy in the mold is quite small when the liquid is cast into the mold smoothly.

In this case, no U-shaped defect appears on the surface of ingot, as shown in Fig. 16(a)~(c). However, if the casting flow is suddenly increased during casting, the liquid not only splashes out of the mold, but also suffers serious turbulence in the mold, as shown in Fig. 15. The U-shaped casting defect and a large amount of shrinkage pores appear on the surface of ingot, as shown in Fig. 16(d)~(f).

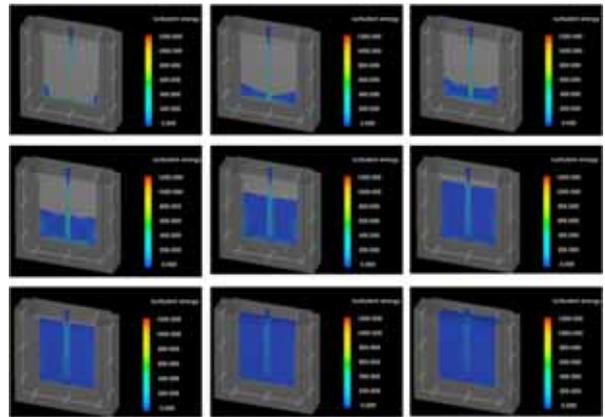


Fig. 14. Simulation results of the turbulent energy of melt cast into the mold with smooth and constant casting flow.

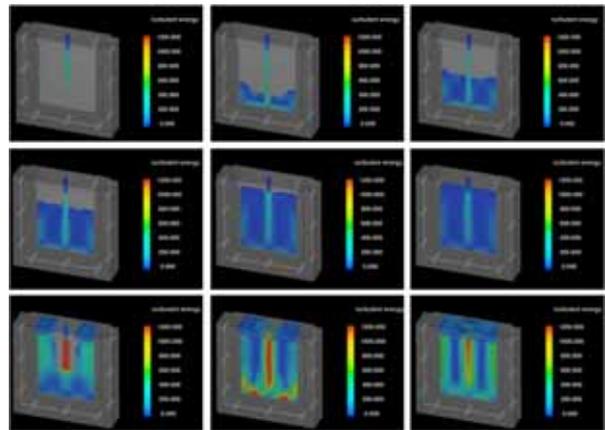


Fig. 15. Simulation results of the turbulent energy of suddenly increasing melt flow.

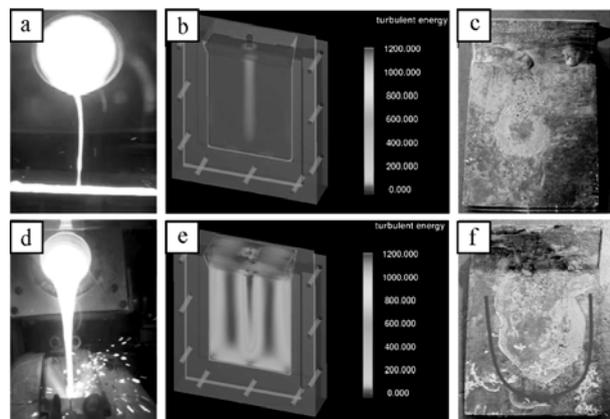


Fig. 16. Relation between the pouring flow of melt and the casting defect on the surface of the ingot.

4. CONCLUSIONS

- (1) The reflection rate of Si-Al thin film increases as the Al content increases; however, Al addition increases the conductivity of Si-Al thin film. Al content of Si-Al thin film should be controlled at around 30wt.% to make Si-Al thin film possess a high reflection rate and insulating property.
- (2) Serious segregation and a large amount of shrinkage pores always appear in the Si-Al ingot because of the large difference between solidus and liquidus temperatures of Si-Al alloy. Therefore, a thin mold should be used to cast Si-Al alloy to speed up the solidification rate of the Si-Al liquid. Casting temperature and mold temperature are the key factors to casting quality of Si-Al alloy.
- (3) As the casting temperature and mold temperature decrease to 1,270 and 50 , respectively, no Al segregation can be observed on the surface of ingot and the percentage of shrinkage pores decreases to 4%. When the casting temperature and mold temperature are higher than 1,300 and 200 , respectively, a large amount of shrinkage pores exists in the ingot, and Al segregation can be observed throughout the ingot.
- (4) A U-shaped casting defect appears on the surface of the ingot if the casting flow is suddenly increased during casting. Al segregation and shrinkage pores

were observed around the U-shaped casting defect.

- (5) If the liquid is cast onto the riser and then flows into the mold along the mold wall, a "hot spot" appears around the riser. After solidification, a large amount of shrinkage pores exists around the "hot spot" because the liquid temperature in this area is too high. To make the liquid flow directly into the mold and to avoid high temperature liquid continually contacting the mold wall, a tundish should be used for casting Si-Al alloy.

REFERENCES

1. J.C. Pan: Industrial Materials Magazine, 253(2008) p. 189.
2. J.C. Pan: Industrial Materials Magazine, 255(2008) p. 193.
3. S. P. Nikanorov: Material Science and Engineering A, 390(2005) p. 63.
4. G. J. Davies: Solidification and Casting, Applied Science Publisher, London, 1984.
5. M. C. Flemings: Solidification Processing, Mcgraw-Hill, New York, 1978.
6. W. G. Winegard: An Introduction to The Solidification of Metals, London, 1964.
7. B. Chalmers: Principles of Solidification, Robert E. Krieger Publishing Company, London, 1964.
8. D. A. Porter: Phase Transformations in Metals and Alloys, Stanley Thornes, UK, 1981.