THE QUANTITATIVE ANALYSIS OF α-AlFeSi RATIO IN A 6063 ALUMINUM ALLOY BILLET BY X-RAY DIFFRACTION

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ABSTRACT The integrated intensity technique of X-ray diffraction (XRD) peak was applied to the estimation of the transformation ratio of α-AlFeSi (α ratio) at the billet surface of 6063 aluminum alloy to study the effect of the α ratio on pick-up formation. As a result, it became clear that the segregation zones affected the pick-up formation. When the α ratio at the billet surface was below 10% in spite of 100% at the billet center, heavy pick-up were observed on the extruded surface. In low α ratio billets, Mg_2Si compounds were formed around β-AlFeSi particles. Thus it was considered that pick-up defects occurred due to peritectic reaction of Al, Mg_2Si and β-AlFeSi at 576 °C.

Keywords: *6063, Al-Mg-Si, AlFeSi, Mg_2Si, pick-up, α ratio*

1. INTRODUCTION

The extrusions of 6063 aluminum alloy are widely used because of its good extrudability and mechanical properties. But its extrudability is sometimes limited by the occurrence of surface defects named pick-up. It has been said that pick-up defects are caused by β-AlFeSi (monoclinic, a = 0.612 nm, b = 0.612 nm, c = 4.15 nm) and/or Mg_2Si particles, and in particular the phase transformation from β- to α-AlFeSi (hexagonal, a = 1.23 nm, c = 2.62 nm) is effective to reduce pick-up [1-2]. Therefore many methods of analyzing α ratio have been developed using various characteristics, such as etching sensitivity of the particles [3], the Si/Fe ratio of the particles, the length of AlFeSi particles, etc. but these methods have some problems to estimate the ratio quantitatively. On the other hand, X-ray diffraction (XRD) patterns have been used to estimate austenite ratio in the steel [4-5]. This method is based on the theory that the integrated intensity of a specific peak depends on the amount of the corresponding phase. In 6063 aluminum alloy billets, the XRD peaks of aluminum, α-, β-AlFeSi and Mg_2Si are appeared. Therefore the α ratio can be estimated quantitatively by measuring the integrated intensities of α- and β-AlFeSi.

In this study, the integrated intensities of XRD peaks were used to estimate the α ratio, and the relationship between the α ratio and pick-up was studied. Furthermore, the mechanism of pick-up formation was discussed.

2. EXPERIMENTAL PROCEDURE

The billets of 6063 aluminum alloy were prepared by DC casting with the composition of Al-0.49%Mg-0.43%Si-0.20%Fe (mass%) and a diameter of 200 mm. The billets were homogenized at 575 °C for 4 to 100 hours. After homogenization, α ratio was estimated at a billet surface. Figure 1 shows the preparation of XRD specimens. First, specimens of 2 mm thick were cut from billet surface and cold-forged to 1 mm thick to flatten and smooth the specimen surfaces. After forging, the specimens were etched to remove surface stain with mixed concentrated acid, and then, XRD studies were carried out. In this study, CuKα radiation was used. Figure 2 shows a example of XRD pattern in this study. The α-AlFeSi ratio Tα (α ratio) were given by

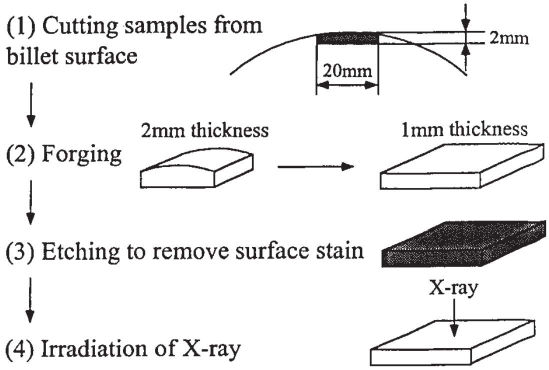

Fig. 1 Schematic model of preparing samples for X-ray diffraction analysis.

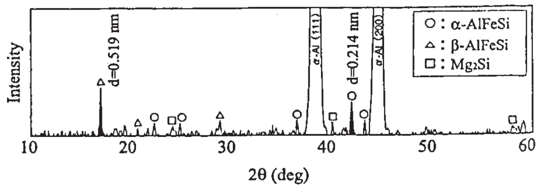

Fig. 2 Typical X-ray diffraction pattern of 6063 aluminum billet surface.

$$T\alpha = I\alpha / (I\alpha + I\beta) \times 100 \ (\%) \qquad (1)$$

where $I\alpha$ and $I\beta$ are integrated intensities of α-AlFeSi at d = 0.214 nm and β-AlFeSi at d = 0.519 nm. The extrusion tests were performed for estimating pick-up. The billets were heated to 420 °C and were extruded at the speed of 45 m/min. The geometry of the extruded section is shown in Fig. 3, and the number of pick-up were counted at the upper surface of the extrusions. Figure 4 shows a SEM image of a pick-up. Pick-up defects are formed with surface tearings and deposits. Thus the pick-up densities were estimated by counting the number of these deposits.

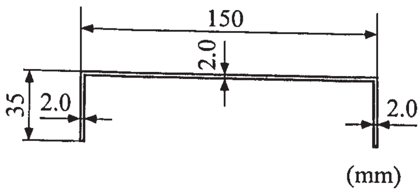

Fig. 3 Geometry of extruded section.

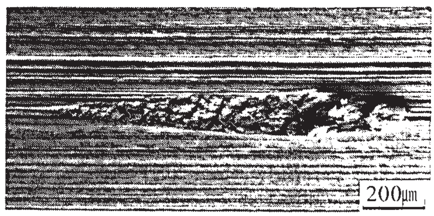

Fig. 4 Appearance of a pick-up defect.

At first a series of billets with various levels of α ratio was used to investigate the relationship between α-AlFeSi ratio and pick-up defects for verifying this quantitative analysis method. Furthermore the effects of segregation zones of the billets on pick-up defects were studied by extruding unpeeled billets with low α ratio and peeled ones. The distribution of elements in segregation zones with various levels of α ratio were investigated using an electron probe micro analyzer (EPMA) to clarify the mechanism of pick-up formation.

3. RESULTS

The relationship between α ratio estimated by XRD and pick-up densities was shown in Fig. 5. It shows that pick-up defects decrease as α ratio increases. In general, it is said that β-AlFeSi particles will cause pick-up defects and phase transformation from β to α is effective to reduce pick-up. The result in Fig. 5 supports the accepted theory and it is considered that the method of estimating the α ratio by XRD is effective in investigating pick-up formation.

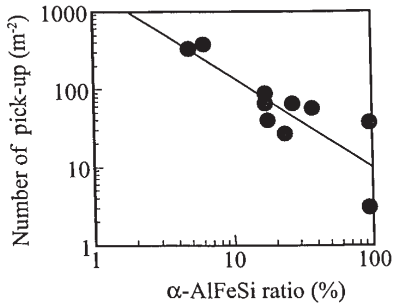

Fig. 5 Relationship between the number of pick-up defects and α-AlFeSi ratio estimated by X-ray diffraction technique.

In a low α ratio billet, the α ratio changed depending on the distance from billet surface shown in Fig. 6. Although the α ratio at the billet surface is below 10% in this specimen, the α ratio is 50% at 1 mm inside and is almost 100% at the center. Figure 7 shows the effect of billet peeling on pick-up defects. After peeling the billet to the thickness of 0.5 mm from the surface, the α ratio is about 15%. The number of pick-up defects in the peeled billet extrusion is about 1/3 of the unpeeled billet one. The relationship between the α ratio and the number of pick-up in peeled billet extrusions lies on the line in Fig. 5. This result shows that the formation of pick-up defects depends on the billet surface quality. For unpeeled billets, the segregation zones have an effect on pick-up. It became clear that estimation of the α ratio must be carried out at a billet surface. Previously developed techniques are difficult, while our XRD technique is easy to estimate the α ratio at billet surface. Thus, the effect of segregation zones on the pick-up defects became obvious by this method.

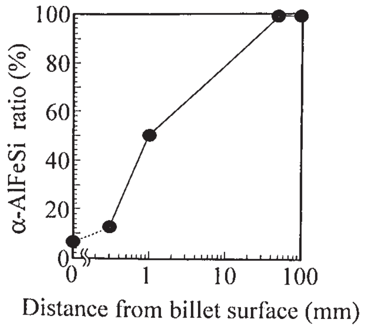

Fig. 6 Effect of distance from billet surface on the α ratio.

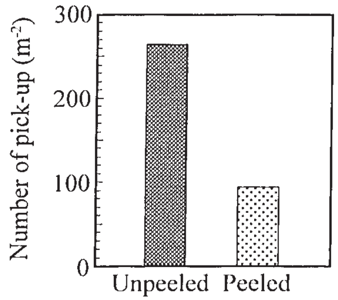

Fig. 7 Effect of billet peeling on pick-up. Billet peeling was carried out to the thickness of 0.5mm from the surface.

4. DISCUSSION

It became clear that the transformation from β- to α-AlFeSi at billet surface, especially at the segregation zones in most cases, reduces the formation of pick-up. But there are two kinds of constituent particles, AlFeSi and Mg_2Si in 6063 aluminum alloy billets. The transformation of AlFeSi particles in homogenization heat treatments became clear, but the behavior of Mg_2Si is not clear. Figure 8 shows the distribution of elements in segregation zones of the billets with various α ratio. From the microprobe X-ray images, magnesium exists with iron and silicon in both homogenized billets with low α ratio and as-cast ones. The Mg_2Si particles formed around AlFeSi constituent particles during solidification. It suggests that these Mg_2Si particles gradually dissolve and β-AlFeSi particles transform to α-AlFeSi simultaneously during homogenization heat treatment.

Figure 9 shows an optical microstructure of the segregation zone. The cell size in this segregation zone is about 100 μm in diameter. In Fig.4, the pick-up deposit was about 100 μm in diameter, so the sizes of the ingot cell and the pick-up deposit are almost same. Furthermore, surface tearings of pick-up defects seem to be melted. Therefore it is suggested that constituent particles in cell boundaries melted during extrusion by the following peritectic reaction in Al-Fe-Mg-Si alloys [6].

$$Al + Mg_2Si + FeSiAl_5 \leftrightarrow liq. + Fe_2SiAl_8 \quad \text{at } 576\ ^\circ C$$

where Fe_2SiAl_8 and $FeSiAl_5$ are known as α- and as β-AlFeSi respectively. In low α ratio billets, it suggests that remelting reaction of cell boundaries takes place at 576 °C because of the existence of Mg_2Si and β-AlFeSi particles. Figure 10 shows the optical microstructures of segregation zones in a low α ratio billet after heat treating at 570 °C, 580 °C and 590 °C for 10 minutes in a salt bath. The marks of melting were observed above 580 °C. It was suggested that the peritectic melting of

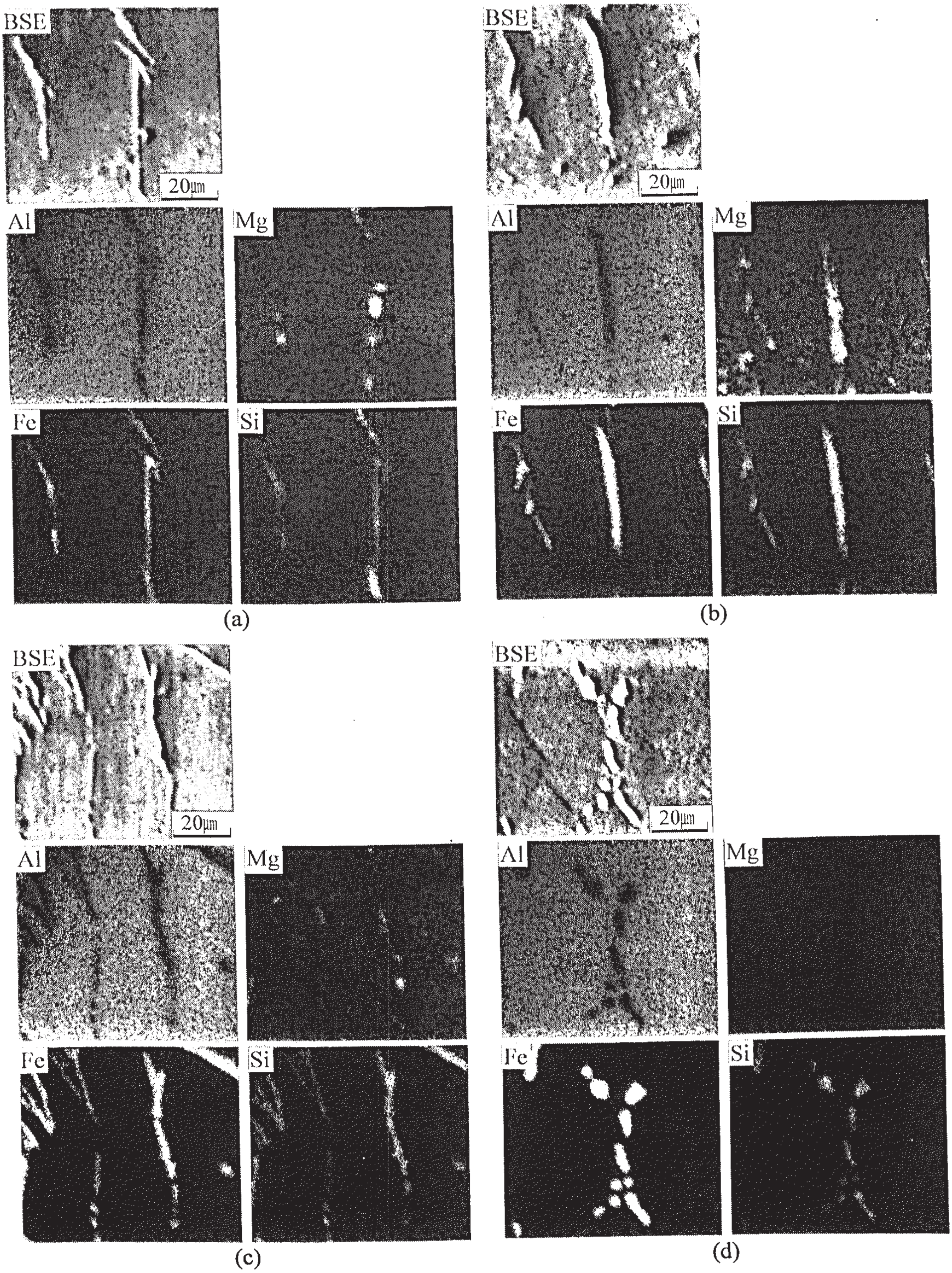

Fig. 8 Distribution of elements in segregation zones of 6063 aluminum billets with various α-AlFeSi ratio. The α ratio were (a) 0% (as-cast), (b) 5%, (c) 20%, (d) 100%.

Al, Mg_2Si and β-AlFeSi at 576 °C was occurred in this low α ratio billet during extrusion. Then, it is concluded that the inhibition of remelting reaction by reducing Mg_2Si and β-AlFeSi particles in cell boundaries results in the reduction of pick-up in higher α ratio billets.

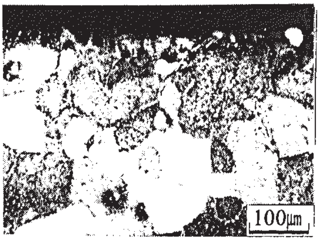

Fig. 9 Optical microstructure of a segregation zone.

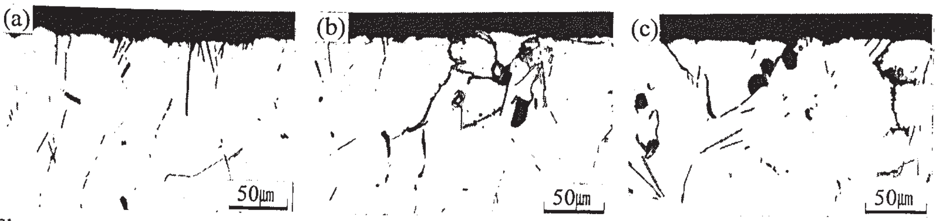

Fig. 10 The marks of peritectic melting of Al, Mg_2Si and AlFeSi in segregation zones of a low α ratio billet. Samples were heat treated for 10 minutes at (a) 570 °C, (b) 580 °C, (c) 590 °C.

5. CONCLUSIONS

(1) The quantitative analysis of α ratio at a billet surface became possible by using a XRD technique.

(2) Segregation zones affected the formation of pick-up.

(3) When the α ratio at a billet surface was below 10%, in spite of 100% at the billet center, heavy pick-up were observed on the extruded surface.

(4) In low α ratio billets, Mg_2Si constituent particles existed around AlFeSi particles.

(5) It is considered that pick-up defects form because of peritectic reaction of Al, Mg_2Si and β-AlFeSi at 576 °C in cell boundaries.

REFERENCES

[1] E. C. Beatty: Pro. 2nd Inter. Aluminum Extrusion Tech. Semi., Vol. 1(1977), 225.

[2] L. R. Morris, F. B. Miner and J. B. Lowe: UK Patent, 1333327.

[3] S. Zajac, B. Hutchinson, A. Johansson and L. -O. Gullman: Mater. Sci. Tech., 10(1994), 323.

[4] A. P. Voskamp: Adv. in X-Ray Analysis, 20(1977), 529.

[5] Carol J. Kelly, M. A. Short: Adv. in X-Ray Analysis, 15(1972), 102.

[6] L. F. Mondolfo: Aluminum Alloys, Structure and properties, Butterworths, London, (1976), 658.