REFINEMENT OF FE-INTERMETALLIC COMPOUNDS BY CALIBER ROLLING PROCESS OF AL-MG-SI-FE ALLOYS

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Abstract

The recycling process of aluminum scraps commonly contains high amount of impurities, especially Fe content which results in degraded properties compared with the primary aluminum production. The refinement of Fe-intermetallic compounds becomes increasingly important to extend the utilization of recycled aluminum. In this study, a thermo-mechanical process of Al-(2.2-2.3)mass%Si-0.9mass%Mg-(1.0-2.0)mass%Fe alloys by caliber rolling process was performed to investigate the refinement of Fe-intermetallic compounds and the mechanical properties. Fine fragmented Fe-intermetallic particles from around 200nm were achieved after 95% caliber rolling. The caliber rolling can effectively improve mechanical properties with good ultimate strength of 345-360MPa and high elongation of 15-25% in comparison with the as-cast 1%Fe specimen with low ultimate strength of 269MPa and low elongation of 1.5%.

1. Introduction

Aluminum alloys have been widely used as light-weight structural materials with good mechanical properties and formability. With the emergency of energy efficiency technology especially in modern vehicles, the utilization of aluminum alloys becomes a good alternative to other conventional engineering materials. Moreover, the recycling process of aluminum scraps is considered as a high energy efficiency process, because the production of recycled aluminum alloys requires only around 3% energy consumption compared with the primary production from bauxite. However, Fe-intermetallic compounds in the recycled aluminum alloys are considered as harmful to mechanical properties and formability, especially the plate-like B-AlFeSi. Recently, severe plastic deformation by caliber rolling (CAROL) has been studied for various materials to produce ultrafine grain structures with good toughness improvement and refinement of intermetallic compounds. Inoue et al. investigated the strain distribution in SM490 steel by finite element analysis and experiment [1]. Mukai et al. observed the refinement of the grain structure of a Mg-Al-Zn alloy by caliber rolling with good improvement of tensile yield stress [2].

In this study, high Fe contents between 1-2mass% (commonly up to 0.1mass% in the commercial aluminum alloys) are intentionally added to the newly proposed Al-Mg-Si alloys in order to investigate the negative effects of Fe impurities in the conventional casting and the subsequently improvement by caliber rolling. Severe plastic deformation by caliber rolling was performed to refine the harmful Fe-intermetallic compounds into more favorable size, shape and distribution in the Al matrix with improved mechanical properties.

2. Experimental Procedure

Table 1 shows the chemical compositions of 3 newly proposed Al-Mg-Si-Fe alloys used in this study. Fig. 1 shows the schematic illustration of the caliber rolling. Aluminum alloy ingots in the size of 53x53x250 mm³ were cast by an induction furnace. Then the ingots were homogenized at 510° C for 2h. After that the ingots were sequentially caliber rolled at 300° C and 450° C up to 95% area reduction as shown in Fig. 2 and Fig. 3. During caliber rolling, the specimens were reheated after 3 passes for 5 minutes to keep the constant rolling temperatures until the final rolling step.

In order to increase the mechanical properties by age hardening, the T6 treatment was performed at 550°C for 1h followed by aging at 180°C for 5h. The characterization was performed with optical microscope, DSC SEM, hardness test and tensile test.

Table 1: Chemical compositions of Al-Si-Mg-Fe alloys (mass%)



Figure 1. Schematic illustration of caliber rolling process.



Figure 2. Caliber rolling machine and specimens with various area reductions.



Figure 3. As-cast specimen in the far left-hand side and caliberrolled specimens with increasing cross section reduction ratios up to 95% in the far right hand side.

3. Results and Discussions

3.1 As-cast microstructures

In the as-cast 1%Fe alloy, long Fe-intermetallic compounds are found in high Fe segregation regions in the center area as shown in Fig.4 a), whereas finer Fe-intermetallic compounds are found in the outer area as shown in Fig.4 b). The amount of the high Fe segregation region increases with increasing Fe content. High amount of Chinese script a-AlgFe₂Si is present in the center area of the 1.5%Fe alloy with fine β -Al₅FeSi in the outer area as shown in Fig.4 c) and d), respectively. Polyhedral a-Al₈Fe₂Si and Chinese script a-Al₈Fe₂Si are found in the center area of the 2%Fe alloy in Fig.4 e). Fine Fe compounds are dominant in the outer area as shown in Fig.4 f), similarly as in the 1% and 1.5%Fe alloys. The type and morphology of Fe intermetallic compounds strongly depend on alloy compositions and solidification condition of the as-cast ingot. Segregation of the Fe enriched region in the center area result in various types and shapes of Fe-intermetallic compounds. On the other hand, high cooling rate in outer area results in fine Feintermetallic compounds. The plate-like shape of B-Al₅FeSi was found as more harmful than α-Al₈Fe₂Si because of its size and shape which can initiate cracking at the interface between the Feintermetallic compound and the Al matrix [3].

DSC heating curves show endothermic peaks of 4 intermetallic compounds: Mg₂Si, Si, β -Al₅FeSi and α -Al₈Fe₂Si, respectively. The amounts of β -Al₅FeSi and α -Al₈Fe₂Si increase with increasing amount of the Fe content. On the other hand, the amounts of Mg₂Si, Si decrease with increasing amount of the Fe content, as a result of reduced Si content which competitively formed by β -Al₅FeSi and α -Al₈Fe₂Si.

3.2 Caliber rolled microstructures

The deformed morphologies of Fe-intermetallic compounds strongly depend on the as-cast microstructures and deformation behaviors. After 95% caliber rolling, the deformed Fe-intermetallic particles in the outer area are well fragmented and distributed in the Al matrix in Fig.6 b). Inoue et al. [1] found that high strain accumulation was generated by high deformation ratios of caliber rolling, especially in the outer area of the specimen. Less fragmentation and distribution of Fe-compounds is found in the center area as shown in Fig.6 a), with less strain deformation compared with the outer area and high segregation of Fe rich region in the center of the as-cast ingot as shown in Fig.4 a), c) and e). The Fe-intermetallic compounds become finely fragmented as shown in the SEM image in Fig.7 a). The smallest size of fragmented Fe-intermetallic particles is about 200nm in the magnified area of Fig.7 a) image in Fig.7 b).

A severe deformed fibrous structure of α -Al grains was obtained along the rolling direction in Fig.8 a). The α -Al grains become apparently fully recrystallized and coarsen after the T6 treatment with the average grain size of 45 μ m as shown in Fig.8 b).



Figure 4. As-cast microstructure of the 1%Fe alloy at a) center area, b) outer area, the 1.5%Fe alloy at c) center area, d) outer area and the 2%Fe alloy at e) center area, f) outer area.



520 530 540 550 560 570 580 590 600 610 620 630 640 650 660 Temperature, °C

Figure 5. DSC curves of the as-cast 1%Fe, 1.5%Fe and 2%Fe alloys.



Figure 6. Fragmentation of Fe intermetallic compounds after 95% caliber rolling of the 1%Fe alloy in a) center area and b) outer area.



Figure 7. SEM image of fragmented Fe-intermetallic compounds in a) 95% caliber-rolled specimen with b) magnified area.



Figure 8. EBSD image of α -Al grain structure: a) as 95% caliberrolled and b) after T6 treatment.

The supersaturated solid solution of Mg and Si can precipitate differently at elevated rolling temperatures which results in various sizes of precipitates and consequently the hardness. Coarser precipitates are found at 300°C and finer precipitates are found at 450°C as shown in Fig.9 a) and b), respectively. However, these precipitates were dissolved into the Al matrix by the solution treatment at 550°C for 1h. Nanosized precipitates of Mg and Si was produced by aging treatment at 180°C for 5h, which is visible by the optical microscope as shown in Fig.10 a) and b).

The hardness testing of the caliber-rolled specimen at 450°C with 95% reduction shows the highest hardness in Fig.11 a). Lower hardness was found at 450°C with 70% reduction and 300°C with 95% reduction, respectively. The hardness of the T6 treated specimens is summarized in Fig.11 b) with slight increase of hardness with increasing amount of Fe content. The precipitation hardening apparently increase the hardness of the caliber-rolled specimens with 43-59HV to the hardness of after the T6 treatment specimens with 134-141HV, even though the α -Al grains become fully recrystallized after the T6 treatment as shown in Fig.8 b).



Figure 9. Microstructures of 95% caliber-rolled specimen at a) 300°C and b) 450°C.



Figure 10. Microstructures of 95% caliber-rolled specimen after T6 treatment at a) 300°C and b) 450°C.



Figure 11. Hardness of specimens after a) caliber rolling and b) T6 treatment.

3.3 Mechanical properties

The improvement of mechanical properties is summarized in Fig.12. The ultimate strength (UTS) of the 95% caliber-rolled specimens (345-360MPa) is clearly higher than that of the 1%Fe as-cast specimen (269MPa). The improvement of elongation of 15-25% in the caliber-rolled specimen is apparently increased compared only 1.5%Fe of the as-cast specimen. The negative effect of large Fe intermetallic compounds is effectively modified by caliber rolling. Moreover, in comparison with the commercial wrought Al-Mg-Si alloy (A6082-JIS) and the commercial cast Al-Si-Mg alloy (A356-ASTM), which commonly contains less than 0.1mass%Fe, the caliber-rolled specimens show both higher strength and elongation. Even though these caliber-rolled specimens contain high amount of harmful Fe- intermetallic compounds. From this result, the caliber rolling is an effective process to modify the harmful Fe intermetallic compounds into strengthening phase.

The strengthening mechanism can be demonstrated by the fracture surface of the tensile test specimen. Brittle fracture surfaces between the large Fe-intermetallic compounds and the Al matrix of 1%Fe as-cast specimen are shown in Fig.13 a) and c) as a result of weak interface and harmful shape of the plate-like β -Al₃FeSi. The crack initiation and propagation occur along the interface between Fe intermetallic compounds and the Al matrix. With the refinement of Fe compounds by caliber rolling, Fe intermetallic compounds becomes finely fragmented and distributed in the Al matrix. Fig.13 b) and d) show finely fragmented Fe particles surrounding by fine dimple fracture of the Al matrix. The fracture surface passing the Al matrix and the refined fragmented Fe compounds, which decelerate the crack propagation and consequently improve the elongation.



Figure 12. Mechanical properties of 95% caliber-rolled specimens after T6 heat treatment.



Figure 13. Fracture surface of 1.0%Fe alloy: a) and c) as-cast, b) and d) 95% caliber-rolled.

4. Conclusions

The refinement of harmful Fe-intermetallic compounds by caliber rolling of high Fe containing Al-Mg-Si alloys (1% to 2%Fe) shows good modification of harmful Fe-intermetallic compounds into more favorable size, shape and distribution of Feintermetallic particle. Consequently, high strength and high elongation of Al-Mg-Si-Fe alloys are obtained by this process.

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