Effect of Microstructural Features on Intergranular Fractures of Al-Mg-Si Alloy Sheets

Y. Himuro¹, Zou Yong², K. Matsuda³, S. Ikeno³, K. Koyama¹ and Y. Bekki¹

¹ Technical Research Division, Furukawa-sky Aluminium Corp., 1, Kiyotaki-Sakuragaoka, Nikko-shi, Tochigi, 321-1443, Japan

 ² Venture Business Laboratory, Toyama University, 3190, Gofuku, Toyama-shi, Toyama, 930-8555, Japan
³ Department of Materials Science and Engineering, Faculty of Engineering, Toyama University, 3190, Gofuku, Toyama-shi, Toyama, 930-8555, Japan

Keywords: grain boundary precipitates, precipitate free zone (PFZ), cooling rate, step-quenching, natural aging, bendability, crack initiation, automotive sheet.

Abstract

The influence of grain boundary precipitates and width of the precipitate free zone (PFZ) on intergranular fractures of natural-aged Al-Mg-Si alloy sheets was investigated. Five types of quenching pattern including step-quenching techniques were carried out after solutionization to control these microstructural features. A lot of precipitates, which resulted from slow cooling and step-quenching at high temperatures, facilitated the initiation of cracks at grain boundaries during bending and tended to decrease bendability. The influence of the PFZ width on intergranular fractures was quite small in this alloy.

1. Introduction

Al-Mg-Si alloys are increasingly being used in automotive body sheet because they show age hardening during the paint bake process. One of the practical problems of using Al-Mg-Si alloys is that cracks sometimes occur on the sheet surface during bending. The bendability of sheets has been investigated from several perspectives including grain size, proof stress, second-phase particles, and shear band [1-4]. It is also considered likely that microstructural control of the grain boundary could improve bendability since the grain boundary is often the site of crack initiation and the propagation path [2, 5].

Particles on the grain boundary and precipitate free zone (PFZ) affect intergranular fractures of age-hardenable alloys such as the Al-Mg-Si system [6]. It is therefore desirable to decrease the particles on the grain boundary, such as second-phase particles and precipitates formed during quenching, to avoid intergranular fractures. Crack nucleation at the PFZ is accelerated with the increased strength that results from aging at high temperatures. The alloy sheets for automotive bodies are usually formed and bent before paint-bake aging, but clusters are formed during natural aging [7]. It is possible that the PFZ that forms during natural aging facilitates intergranular fractures.

In this study, cooling-rate control and step-quenching experiments were carried out to control the grain-boundary precipitates and PFZ width. The influence of these microstructural features on intergranular fractures during bending is discussed.

Cold-rolled sheets, 1.0-mm thick, were used in this study. The chemical composition of the alloy is given in Table 1.

Table 1: Chemical composition of the alloy.

Element	Si	Fe	Cu	Mg
Mass %	0.99	0.11	<0.01	0.60

The specimens were solution-treated in a salt bath at 540°C for 40s and then quenched with two spray guns. The normal and step-quenching patterns shown in Figure 1 were obtained by controlling spraying pressure and time. For normal quenching, the test pieces were continuously cooled from the solutionization temperature to room temperature. The average cooling rate from 540 to 100°C was about 44, 15 and 8°C/s (referred to as NQ-1, NQ-2, and NQ-3, respectively). For step-quenching, spray cooling was carried out from 540°C to about 100 or 200°C at the same cooling rate as for the NQ-1 pattern, and then stopped. These specimens were naturally air-cooled to room temperature at a cooling rate of less than 2°C/s (referred to as 100SQ and 200SQ, respectively). These specimens thus obtained were naturally aged at room temperature (about 20°C) for 1 week. This aged state is referred to as the T4 condition in this paper. Since the average grain size is about 50 μ m for all the specimens, the effect of differences in grain size on intergranular fractures was disregarded in this study.



Figure 1: Normal and step-quenching patterns.

The tensile test was carried out using JIS5 tensile specimens parallel to the rolling direction. The bending test was conducted in two steps: the specimens were stretched under an applied elongation strain of 15% using a tensile machine, then bent through 180 degrees with an inner thickness of 1.0 mm. Cross-sections, surface morphology, and fracture surfaces of the bent specimens were examined using optical microscopy (OM) and scanning electron microscopy (SEM). Particles on grain boundaries and PFZ structures were observed using a JEM-3010 electron microscope (TEM) operated at 200 kV. Thin foils for TEM examination were prepared by jet-polishing in a solution of methanol:nitric acid (4:1). The PFZ structure was examined after aging the T4 specimens at 170°C for 4 h (T6 condition) since this was difficult to observe in the T4 condition.

3.1 Microstructure

Several particles were dispersed on the grain boundaries of specimens in the T4 condition. TEM bright-field images of the grain boundaries are shown in Figure 2. Energy dispersive spectroscopy (EDS) showed that these particles were mainly magnesium silicides. The particles could be roughly classified into two types: very large particles of over 100 nm, and small ones of around 20-50 nm. The former were observed in all the specimens and the latter were more prevalent in the NQ-2, NQ-3, and 200SQ specimens. It is probable that the large particles formed, or did not dissolve, during the solution treatment at 540°C, and the small ones precipitated on subsequent quenching due to slow cooling. Even with rapid quenching, interruption of the cooling at about 200°C also resulted in precipitation on the grain boundaries. These results suggest that precipitation on the grain boundary during quenching can be suppressed by rapid cooling to at least 100°C.



Figure 2: TEM bright-field images showing particles on grain boundaries of quenched specimens in T4 condition.

The PFZ width varied according to the quenching process used after the solution treatment. A typical PFZ structure of a specimen aged at 170°C for 4 h is shown in Figure 3. Fine precipitates, presumed to be β '-Mg₂Si phase, can be observed in the grains, and PFZ structure are clearly seen near the grain boundary. The PFZ widths of the NQ-1, NQ-2, NQ-3, 100SQ, and 200SQ specimens were about 60, 110, 160, 90, and 80 nm, respectively.

The following points are worth noting: (i) the PFZ width showed a tendency to widen with a decrease in the cooling rate, and (ii) the PFZ widths of the step-quenched specimens were larger than that of the NQ-1 specimen. The formation of the PFZ is thought to be

responsible for the depletion of vacancies and solute atoms around the grain boundary [8]. It appears that slow cooling and step-quenching decreased excess vacancies. Precipitation during cooling can cause a shortage of Mg and Si atoms near the grain boundary. These factors are presumed to affect the width of a PFZ adjacent to the grain boundary. It is interesting to note that the PFZ was wider for the 100SQ specimen than for the NQ-1 specimen, even though there was little difference in the quantity of grain-boundary precipitates. This suggests that the PFZ width depends mainly on the vacancy concentration in this alloy.



Figure 3: PFZ structure of NQ-3 specimen aged at 170°C for 4 h.

3.2 Mechanical Properties and Bendability

The results of tensile tests of the normal- and step-quenched specimens in the T4 condition are shown in Table 2. It has been reported that the 0.2% proof stress and elongation affect bendability [2, 5]. However, these properties are not greatly influenced by the quenching pattern and in this study, little consideration is given to these factors in discussing bendability.

Table 2: Mechanical properties of the quenched samples

Quenching pattern	NQ-1	NQ-2	NQ-3	100SQ	200SQ			
0.2% proof stress (MPa)	125	120	118	119	119			
Tensile strength (MPa)	244	241	239	237	236			
Elongation (%)	33.6	33.5	33.5	32.8	32.9			

Bendability and crack morphologies were examined using OM and SEM. Figure 4 shows cross-section micrographs of the bend-tested specimens. Bendability has a tendency to deteriorate with a decrease in cooling speed and an increase in step-quenching temperature.



Figure 4: Cross-section micrographs of bent specimens.

It can be seen that the cracks did not propagate along the grain boundaries preferentially, but grew along the direction of approximately 45 degrees to the tension surface of the bent sample. The fracture surface morphology of an NQ-3 specimen is shown in Figure 5.

There are a large number of dimples, which were elongated parallel to the fracture surface. These observations indicate that the cracks mainly grew through a transgranular mode and that shear stress affected crack growth during bending deformation.



Figure 5: Fracture surface of NQ-3 specimen.

To clarify the initiation of the cracks, the surface morphologies of the pre-strained and bend-tested NQ-3 specimens were observed. The tension surface became rougher with deformation since the preferred deformation direction differed depending on the grains. As a consequence, the grain boundaries can be clearly seen in the tension surface of the stretched specimens, as shown in Figure 6 (a). Small cracks along the grain boundaries were often observed after the bending test; typical examples are shown in Figure 6 (b).

In grains, there are some microvoids at the interface between the second-phase particles and the matrix, where the particles are mainly Fe-rich phases. Figure 6 (c) shows a macro crack observed on the surface. The macro crack shows a mixed structure of intergranular and transgranular fracture modes. These observations suggest that the tensile and subsequent bending deformation induced small cracks along the grain boundaries and cavities, and that propagation through these cracks and cavities resulted in the formation of the macro crack.



Figure 6: Tension surface morphologies of NQ-3 specimen (a) after tensile deformation, and (b, c) after bending deformation.

4. Discussion

Cracks seem to appear and grow on bending according to the following processes: small cracks at the grain boundaries and cavities occur on the tension surface, they connect, and then grow along the shear direction mainly through a transgranular fracture mode. Slow cooling and step-quenching at high temperatures resulted in the precipitation of magnesium silicide particles on the grain boundaries during quenching. Increases in the

number of these particles presumably facilitated the initiation of cracks at grain boundaries. This is probably why there was a loss of bendability with a decrease in the cooling rate and an increase in the step-quenching temperature.

The PFZ in the T4 condition may affect intergranular fractures during bending. The PFZ structure was examined after the T6 treatment since it was difficult to observe it in the T4 condition in this study. It is thought that Si-vacancy clusters are formed during natural aging and that the clusters do not act as nucleation sites for the precipitation of β '-Mg₂Si [7]. Consequently, the PFZ width in the T4 condition might not correspond with that in the T6 condition. However, since the vacancy concentration strongly affects the formation of Si-vacancy clusters during natural aging as well as the PFZ formation during T6 aging, it is likely that there is no great difference between the width of the PFZ in the T4 and T6 conditions. Based on this assumption, the effect of the PFZ on intergranular fractures is discussed as follows. Comparing the NQ-1 and 100SQ specimens showed that the PFZ was widened without increasing the precipitates on the grain boundaries by stepquenching. However, the bendability of the 100SQ specimen hardly differed from that of the NQ-1 specimen. This suggests that the PFZ width in the T4 condition does not have a significant effect on intergranular fractures. It is possible that the effect of PFZ width on intergranular fractures is increased by natural aging for long periods, but further study is needed to clarify this.

5. Conclusions

- 1. Slow cooling and step-quenching at high temperatures after solution treatment caused the precipitation of magnesium silicide on grain boundaries and widening of the PFZ.
- 2. Flex cracks grew along the shear direction mainly through a transgranular fracture mode, but the cracks were presumed to begin at grain boundaries and cavities on the tension surface of bent specimens.
- 3. The dominant factor in intergranular fractures in the T4 condition appeared to be the quantity of grain-boundary particles.

Acknowledgments

This work was performed under the management of the Japan Research and Development Center for Metals (JRCM) as a part of Japan's National Project for "Nanotechnology Metal Project" supported by the New Energy and Industrial Technology Development Organization (NEDO).

References

- [1] Y. Takagi, T. Masuda and T. Sakurai, Abstracts for the 97th Conference of Japan Institute of Ligth Metals, 177-178, 1999.
- [2] M. Saga, M. Kikuchi, Y. Zhu and M. Matsuo, Proceedings of ICAA-6, 425-430, 1998.
- [3] M. Asano, H. Uchida and H. Yoshida, J. Jpn. Inst. Light Metals, 52, 448-452, 2002.
- [4] A. Hibino, T. Muramatsu, M. Saga and K. Takata, J. Jpn. Inst. Light Metals, 53, 534-541, 2003.
- [5] G. Itoh, T. Suzuki and K. Horikawa, Materials Science Forum, vol 396-402, 1193-1198, 2002.
- [6] A. K. Vasudevan and R. D. Doherty, Acta Metall., 35, 1993-1219, 1987.
- [7] K. Yamada, T. Sato and A. Kamio, Materials Science Forum, vol 331-337, 669-674, 2000.
- [8] S. Fujikawa and K. Hiraga, J. Jpn. Inst. Met., 10, 667, 1971.