Color Metallography of Characteristic Microstructure in High-Speed Twin-Roll Cast Al–Mn–Si Alloy Strip Using Week’s Reagent

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A color metallography using Week’s reagent was employed to investigate the characteristic microstructure of Al–Mn–Si alloy strip fabricated by high-speed twin-roll casting. The microstructure of the strip consists of two components: solidified shells and a central band. By Week’s reagent etching, the colorful microstructure was obtained, and doughnut-like patterns were observed in the globular grains. Based on the presence of the patterns, the globular grains were divided into two types: Type-I and Type-II. Type-I grains exhibited the core-like structure. On the other hand, Type-II grains had no color contrasts in the grain. SEM-EDS analysis of Type-I grains revealed the high correlation between the obtained color and micro-segregation of Si. TEM and STEM analyses confirmed the formation of an amorphous film on the surface of Al substrate by the etching. The thickness of the film and the roughness of the Al substrate under the film were different from location to location. The local change of the film’s features resulted in the different color in the optical microscopic image. Based on the microstructure observation, the origin of globular grains observed in the central band in the Al–Mn–Si alloy strip was discussed in detail. [doi:10.2320/matertrans.L-M2020856]

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1. Introduction

1.1 Week’s reagent for aluminum alloys

Color etching is a useful technique to characterize the microstructures of various metals and alloys. “Week’s reagent” was developed by Weck and Leistner in 1980s,1 and it is known to be very sensitive to the micro-segregation of solute atoms in aluminum matrix. This reagent contains 4 g of KMnO4, 1 g NaOH and 100 ml of distilled water, and the color images of the microstructure can be obtained by etching. Gao et al.2,3) reported that the micro-segregations of Ti and Si occurred during solidification of die-cast A356 aluminum alloys can be revealed vividly by this color etchant. Suárez-Peña et al.4) applied Week’s reagent for Al–Si 12 alloy (Al–12.9 mass% Si–0.82 mass% Fe–0.2 mass% Mn), in which α-Al were given even from a small difference between the central and peripheral areas. The mechanism of the etching process is explained by the following chemical reaction:

\[
\text{Al} + \text{MnO}_4^- + 2\text{H}_2\text{O} \rightarrow \text{Al(OH)}_4^- + \text{MnO}_2
\]

This chemical reaction was proposed based on the studies of aluminum’s chemical activity5,6) and permanganate conversion coating on 2024 Al alloy.7) The formation of MnO2 films is based on the above reaction. The color differences by Week’s etching are considered to be due to the micro-segregation of alloying elements in the Al matrix. It is expected that Week’s reagent can be a good option to reveal the characteristic microstructure and examine the segregation for the Al based strips fabricated by the high-speed twin roll casting.

1.2 High-speed twin-roll casting (HSTRC)

High-speed twin-roll casting (HSTRC) is one of the prospective near-net-shape casting techniques to fabricate thin aluminum alloy strips from the molten alloy directly at a high speed (60~120 m/min).8–11) The capital investment and operating cost for HSTRC are lower than the conventional direct chill (DC) casting due to the elimination of an intermediate hot-rolling process. Production of the strips by HSTRC has various advantages such as microstructure refinement of the cast strips,12–18) enhancement of solubility of alloying element and improvement of mechanical properties of the final cold-rolled and annealed sheets.19,20)

The microstructure of the HSTRC aluminum alloy strips characterized by the mushy-type solidification usually contains two main components: the solidified shells grown from the roll surfaces and the central band structure sandwiched between them.10) The band structure is rarely observed in other cast products, and is a characteristic feature of the HSTRC strips. The band structure including many globular grains is often observed at the mid-thickness region of the cast strip. Kim et al.17,18) reported that the formation of the central band is partly due to the condensation of the primary Al crystals which are separated from the solidifying shells on the roll surface, and the broken dendrite branches of the growth front of the solidifying shells. The formation of the band structure is inevitable in the alloys which have a wide freezing temperature range. Reduction of the band structure is important in order to improve the quality of the cast strips, however, there have been limited researches on the formation mechanism of the band structure.

The micro-segregation of the alloying elements can provide many useful information for investigating the solidification mechanism. It is known that the colorful microstructure of the cast Al–Mn–Si strips can be obtained by using Week’s reagent. The previous studies2,3) revealed that the color difference was given even from a small composition difference of the alloying elements. Therefore, it is expected that color metallography by Week’s reagent provides useful information concerning the solute segregat-
tion manner in the central band and its formation behavior during the strip casting.

In the present work, the characteristic microstructure of Al–Mn–Si strips fabricated by HSTRC was characterized by using the color etching technique with Weck’s reagent. Various kinds of observation and analyses were carried out to investigate the topography, morphology and chemical composition of the etched samples. The correlation between the color difference and micro-segregation of the alloying elements was examined in detail, and the formation behavior of the central band as well as the origin of the globular grains in the central band were discussed.

2. Experimental Procedures

2.1 Material, casting and heat-treatment processes

In the present work, the Al–Mn–Si aluminum alloy was used. Chemical composition of the alloy is shown in Table 1. The ingot was supplied by Mitsubishi Aluminum Co. Ltd. The ingot was re-melted and cast into a strip by using the vertical-type high-speed twin-roll caster. The schematic diagram of the caster is shown in Fig. 1. The caster consists of a pair of pure copper roll, series of springs and a nozzle/side-dam component. The size of the rolls is 300 mm in diameter and 100 mm in width. Any kind of lubricant on the roll surface and melt (or solidified shells). The nozzle is covered with heat-insulating materials.

The alloy strips were fabricated by HSTRC. About 2.5 kg of the alloy ingot was re-melted in a clay-graphite crucible by using an electric resistance furnace. The melt was degassed by the argon gas bubbling for 20 min before pouring into the nozzle. During the casting, the melt was solidified on both roll surfaces to form solidification shells. The solidification shells were introduced to the roll gap by the roll rotation and pressed to form the strip. The details of the casting condition are described in Table 2.

In order to confirm the solute segregation in the crystal grains by the precipitation behaviour, some samples collected from the cast strip were heat-treated at 450°C for 8 hours in the salt bath.

2.2 Microstructure observation and chemical analysis

Specimens used for microstructural observation were sectioned from the stable thickness part of the cast strip. The specimens were mounted in a resin and polished with grinding papers from 120# until 4000# and diamond paste (6, 3, 1 µm). The final polishing was done by using a colloidal silica-OPS solution. The polished specimens were etched with Keller’s solution (HF: 1 ml, HCl: 1.5 ml, HNO₃: 2.5 ml, and distilled water: 95 ml), and Weck’s reagent (NaOH: 1 g, K₂MnO₄: 4 g, and distilled water: 100 ml). The specimens were immersed in Weck’s reagent for 12 s at about 25°C. For the alloy used in the present study, 12 s is the best condition to obtain a good color contrast. The microstructure of the specimens was examined by using an optical microscope-OM (OLYMPUS, BX51M).

The JEOL 7000 FE SEM was used for microstructural observation and chemical analysis. The etched surface was analyzed by Secondary Electron Image (SI-image). EDS mapping and point analyses were used to analyze the chemical composition. The accelerating voltage was set at 12 kV for SI mode and 15 kV for EDS mode.

AFM5100N Compact General-Purpose Atomic Force Microscope (HITACHI) and SFT-4500 Nano Scan Microscope (SHIMADZU) that combined a laser microscope (LSM) and scanning probe microscope (SPM) were applied to investigate the surface topography of etched samples. These microscopes contain the optical microscopes for the accurate selection of the target area. The surface average roughness was calculated and represented by the Ra value.

The JEOL JEM-2010F microscope (200 kV) was used for TEM and STEM observation. In the beginning, carbon (C) and tungsten (W) were deposited on the etched surface to protect the film formed by the etching. Thickness of the C coating layer was about 80 nm, and that of W coating layer was about 600 nm. Focused ion beam (FIB – FB 2100 Hitachi High-tech) was used to section the samples for TEM and STEM observation. Energy dispersive X-ray Spectroscopy (EDS) equipped in STEM was employed to analyze the composition of the film.

Table 1 Chemical composition of the alloy used in this study (mass%).

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Al</th>
<th>Mn</th>
<th>Fe</th>
<th>Si</th>
<th>Other</th>
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<tr>
<td>Al-Mn-Si</td>
<td>Bal</td>
<td>1.130</td>
<td>0.003</td>
<td>0.920</td>
<td>0.002</td>
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</table>

Table 2 Casting conditions for HSTRC sample.

<table>
<thead>
<tr>
<th>Casting temperature</th>
<th>675 °C (+15 °C superheating)</th>
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</thead>
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<tr>
<td>Solidification length</td>
<td>100 mm</td>
</tr>
<tr>
<td>Applied force</td>
<td>60 kN</td>
</tr>
<tr>
<td>Roll speed</td>
<td>60 m/min</td>
</tr>
<tr>
<td>Initial roll gap</td>
<td>1 mm</td>
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</tbody>
</table>
3. Results and Discussion

3.1 Strip profile and microstructure observation of strip

About 3 m-long continuous strips were successfully fabricated by HSTRC. Figure 2 shows the thickness change of the strips along the casting direction. The strip thickness increased gradually and then became stable. The average thickness in the stable region was about 2.6 mm. The samples for microstructure observation were collected from the stable thickness area.

Small size samples which were fitted for the standard grinding holder were extracted from the central area of the cast strip along the casting direction. They were polished and etched by both Keller’s and Week’s reagents. Figure 3 shows the microstructure for the half thickness of the cross-section. The left is the strip surface and the right are the mid-thickness area of the strip. The microstructure consisted of two main components: the solidified shell region grown from the roll surface and a central band region. In the solidified shell region, the microstructure changed gradually along the thickness direction. At the near-surface region, the cellular structure with very fine dispersed particles was detected. The cellular structure gradually changed to the equiaxed dendrite structure in the quarter of the thickness region. The microstructure in the mid-thickness area of the strip (at the right-hand side of the picture) was totally different. The structure consists of fine globular grains and eutectic solidified region appeared as a dark contrast. No voids and cavities were observed in this area.

Enlarged micrographs of each region in Fig. 3 are shown in Fig. 4(a), (c) and (e), respectively. They were etched with Keller’s reagent. Columnar crystals with cellular dendrite structure at the near-surface, and equiaxed grains with dendrite structure at the quarter of the thickness are observed. The central band thickness was about 2% to 20% of the total strip thickness (i.e., central band thickness was not constant along the casting direction), and showed a characteristic microstructure consisting of globular grains. The microstructure was observed by using Week’s reagent. The results are shown in Fig. 4(b), (d), and (f). No kinds of optical filters were used for observation in this case. By using the Week’s reagent, grain boundaries and arrays of dispersed particles were visualized clearly. The most important finding is that we can observe a large number of doughnut-like patterns in many globular grains located in the central band (see the arrows in (f)). It should be mentioned that the same doughnut-like patterns can also be seen in (b) and (d), as shown by the arrows. This seems to suggest that the globular grains with the doughnut-like patterns were trapped in the growing cellular and equiaxed dendrites during the roll casting.

Figure 5(a) and (b) are high magnification images of the central band region etched with Keller’s reagent and Week’s reagent, respectively. No contrast difference is observed in the globular grains in Fig. 5(a), while the doughnut-like patterns are visible in globular grains in Fig. 5(b). We can also observe some grains without these patterns in Fig. 5(b). Based on the appearance of the pattern, the globular grains can be divided into 2 types: Type-I and Type-II. Type-I grains with the characteristic patterns are marked by the arrows in Fig. 5(b). Type-II grains with no color contrast in the grain are shown in the same picture. It seems to be possible that the color difference produced by Week’s etching results from the segregation of alloying elements. Therefore, in the next section, the chemical composition of the Type-I grain is carefully investigated.

3.2 Solute distribution in the Type-I globular grain

Figure 6(a) shows an optical micrograph of one of the Type-I globular grains which was located in the central band. A characteristic core-like structure is observed. A light-yellow round area is traced with a dark-brown ring. Figure 6(b) is the close-up of the area in the red box in (a). Solute distribution was analyzed by SEM-EDS mapping for the area of (b). Figure 6(c) and (d) show the Si and Mn distribution. The attached color bar shows the difference of the concentration as the change of X-ray intensity. In the Si mapping, the color change from black to pink represents the increasing of Si concentration. Inside the core region, the observed color was almost blue, while more green color was observed in the outer region. This result indicates that Si concentration is lower in the core region. For Mn mapping, there was a small change of Mn over the whole grain area as the red color appeared uniformly on the Al matrix. It indicates Mn uniformly distributed in the whole grain area.
Solute concentration was also investigated by SEM-EDS point analysis. Figure 7(a) shows the position of the point analysis. Figure 7(b), (c) and (d) show the EDS pattern for three different positions: point 1, 2 and 3. The Si-Kα peaks observed at point 1 and 2 were relatively weak. The values of concentration for Mn and Si in each position are shown in Table 3. Mn concentration was about 0.5% for all three positions. In contrast, Si was detected only at the point 3, the concentration was about 0.2%. These results are consistent with the result obtained by EDS mapping. Mn distributed uniformly in the whole area of the globular grain, while Si was only observed at the outer area. This result suggests that the distribution of Si may control the color observed after Weck’s etching. Generally, the color obtained in an optical microscope results from the interaction between the light and surface of the samples. Therefore, the surface condition of the etched sample was also investigated.

3.3 Surface of the sample etched with Weck’s reagent

The surface morphology of the etched sample was analyzed by SEM-SI. Figure 8(a) shows the SEM image of one of the Type-I globular grains. The surface condition was different in the core and outer regions. Both surface erosion and formation of some surface film by etching may result in the change in surface roughness. It should be mentioned that, however, only the effect of surface erosion of the Al substrate can be detected by the present SEM analysis. Figure 8(b) and
3.4 Surface topography of etched samples by laser microscopy and AFM

Surface of the etched sample was also investigated in detail by both laser microscopy and AFM. Figure 9 shows the (c) show the enlarged image of the core (region 1) and the outer (region 2). Many deep and elongated cracks are observed in region 1. In region 2, the surface is smoother and no cracks are observed in the outer region.
surface topography obtained by laser microscopy for Type-I grains etched with Weck’s reagent. Three kinds of information were indicated here: laser intensity, color and surface height profile. The laser intensity represents the difference in reflection intensity of laser from location to location. A bright contrast means the high intensity of the reflected laser. A large reduction in the reflection intensity was obtained in the core, as shown in Fig. 9(a). Figure 9(b) shows a color taken by laser microscopy. The represented colors are consistent with those obtained by OM in Fig. 6(a) very well. The core shows a strong yellow color. A number of cracks are observed in the core, as well as the SEM picture (Fig. 8(b)). The edge of the core represents a dark brown color, and this area corresponds to the region of the lowest laser intensity in Fig. 9(a). The outer area of the core is light yellow and it corresponds to the highest intensity area in Fig. 9(a). Figure 9(c) shows the surface height profile. This profile indicates that the height is different among the inside-core, edge of the core, and outside the core. The area outside the core which has the light-yellow color is higher than the one inside. The highest position is observed at the edge of the core, and it has a dark brown color. In order to highlight the difference in surface height, the 3D picture was constructed from the surface profile data.

Figure 10 shows the 3D surface profile in the globular grain. In order to express the correlation between the locations and the observed color clearly, the optical color image of the concerned grain in 2D is added on the upper right of Fig. 10. The intense yellow area corresponds to the core, while the light yellow area corresponds to the outer region. The maximum relative height is observed at the edge of the core and the grain boundary. The lowest relative height is observed in the core. The average roughness ($Ra$ value) was measured in the area marked by red boxes and arrows. $Ra$ values are 31 nm inside the core and 94 nm at the edge. The value of the outer region is about 3 nm, which is ten times lower than that of the inside the core. These results indicate that the surface of the core region is relatively rough, but that of the outer region is quite smooth. The highest $Ra$
value is obtained at the edge of the core, which is the transition zone between two regions.

In contrast, Fig. 11 shows the surface topography obtained by AFM. The selected area of this image is the same as the one for the laser microscopy images shown in Fig. 9 and 10. The optical color image of the grain is also pasted on the upper right side of Fig. 11(b). Figure 11(a) and (b) show the overall views of the grain. Figure 11(c) is the scanned images for the $2 \times 2 \mu m^2$ area inside the core region (region 1 in Fig. 11(a)), and Fig. 11(d) is the $2 \times 2 \mu m^2$ area of the outer area (region 2 in Fig. 11(a)). The surface topography of the grain detected by AFM was clearly different from the one obtained by laser microscopy. The AFM result shows that the whole surface of the grain is relatively flat. Only a small difference in roughness is observed between the inside and outside of the core. The $Ra$ for the area inside the core is 14.56 nm and that of the outer region is 4.56 nm. Ripple marks are also observed in Fig. 11(c) and (d), which were observed inside the core region in the SEM and laser microscopy images.

For laser microscopy, the incident laser can be reflected by both the top surface of the sample and the interface between substrate and the film. However, it is reported that the image of laser microscopy reflected only the profile of the interface between the substrate and film in case that the film formed on the substrate is very thin ($<1 \mu m$). Therefore, when the surface film is very thin, the obtained surface profile as shown in Fig. 9 and 10 represent a topography of the interface between the substrate surface and the bottom surface of the film. In contrast, it is possible that the surface topography obtained by AFM corresponds to the profile of only the top surface of the thin film, since the surface of the etched grain was scanned by using the cantilever.

3.5 Microstructural observation and compositional analysis of the film by TEM and STEM/STEM-EDS

Formation of the thin film was confirmed on the etched surface of the A356 alloy with Week’s reagent in the previous study. It is expected that the film also formed on the etched surface in the present case. The existence of the film and its properties were investigated by TEM and STEM. Figure 12(a) and (b) show the TEM images of the cross-section of the film in the core and outer area of the grain, respectively. The shape of the film is indicated by white arrows. The films have different thickness and morphology in each area. The film thickness in the core area is about 170 to 200 nm, and fine columnar grains grown along the thickness direction in the film are observed as shown in Fig. 12(a). The film thickness in the outer area is thinner than that in the core area as shown in Fig. 12(b). The diffraction pattern of the film was taken by nano-beam electron diffraction (NBD). The beam size was about 10 nm. The diffraction pattern revealed that the film is amorphous.

Figure 13 is the cross-section of the film observed by STEM. Figure 13(a) shows a relatively low magnification image of the cross-section of the film. In this view, the lower-side of the image corresponds to the Al substrate, and the upper-side corresponds to the C and W coating layers. The right-hand and left-hand sides in the image corresponds to the core and outer regions, respectively. The thick film is observed in the core region, while the thin film is observed in the outer region. It is important to note that, the film’s surface is relatively flat, and it is consistent with the result obtained by AFM. The details of the films are shown in Fig. 13(b) and (c). The location of the film is highlighted by the white dash lines. The thickness of the film is about 170 to 200 nm in the core, whereas it is about 20–30 nm in area outside of the core.
Chemical composition of the film in Fig. 13(b) and (c) was analyzed by STEM-EDS. The results are shown in Fig. 14. It was revealed that both thick and thin film consist of Mn and O, and this indicates the formation of manganese oxide.

### 3.6 Relationship between the film formation and micro-segregation of Si

The formation of the film consisting of Mn and O was confirmed by TEM and STEM/STEM-EDS. The formation process of this kind of film is related to the term “conversion coating” in coating industry. In the case of Al alloy, there are two popular processes: chromate conversion coating (CCC) and permanganate conversion coating (PCC). In PCC process, Al is consumed to produce a manganese oxide film on the substrate and it is considered to be very similar to the...
present etching by using Weck’s reagent. The thickness and morphology of the film changed depending on the location. These results show a good agreement with the findings in the previous works for A356 alloy containing Ti. In the case of A356 alloy, Ti segregation in the Al grain strongly affected the thickness and morphology of the film.

Figure 15 shows a schematic diagram to explain the coloring mechanism. Figure 15(a) shows an obtained color in Type-I globular grain under an optical microscope. Figure 15(b) shows the change in Si concentration, film thickness and Al substrate roughness in the grain. Figure 15(c) is a schematic diagram of the cross-sectional view near the surface. Si concentration was low inside the core, where the thick film was formed (Fig. 12(a) and 13(b)). The surface of the Al substrate, or the boundary between the surface of Al substrate and the bottom surface of the film was smooth. The thin film was formed in area the outer area of the grain with high Si concentration. In this case, the interface between the substrate surface and the bottom surface of the film was relatively smooth. In addition, the top surface of the film was smooth. The film thickness, the morphology of the surface film and roughness of the substrate were different from location to location. These differences may provide various kinds of combination of the interference of reflected light, the diffraction and the absorption of a certain wavelength of manganese oxide or any two of these three factors, and result in the characteristic color difference under the optical- and laser-microscopical conditions. This idea is considered to be reasonable, given the experimental results in the previous works.3,19

3.7 Inhomogeneous distribution of precipitates caused by micro-segregation of Si

A partitioning coefficient of Mn in Al is about 0.95 according to the equilibrium phase diagram of Al–Mn system.20 The partitioning coefficient of Si in Al is small and it is about 0.13 according to Al–Si equilibrium phase diagram.20 This may result in a more severe micro-segregation of Si than Mn during solidification. By SEM-EDS analysis, it was revealed that Mn uniformly distributed over the whole area of the grain. Therefore, the local difference in thickness and morphology of the film is considered to be due to the micro-segregation of Si.

In order to confirm the micro-segregation of Si, some samples collected from the cast strips were heat-treated at 450°C for 8 h and the resultant precipitation structure was examined. Figure 16 shows the microstructure of the central band after the heat treatment. Precipitates are observed as dark pits here. The white arrows indicate the Type-I grains exhibiting the doughnut-like patterns by the Weck’s reagent etching. In contrast, the black arrows indicate the Type-II grains with no color difference by the Weck’s reagent etching. The important finding is no or less amount of precipitation occurred in the core region of the Type-I globular grain. It is known that, in Al–Mn–Si ternary alloys, Si distribution in the Al matrix controls the precipitation manner and the formation of Al–Mn base precipitates is accelerated by the addition of Si. The no or less amount of precipitates inside the core of the Type-I grains indicate the low Si concentration in this region. These results suggest that there is a strong relationship between Si distribution in the Al grain, morphology of the etched surface, characteristics of the surface film and the color revealed by the Weck’s reagent etching.

3.8 Application of Weck’s reagent etching to investigate the formation mechanism of the central band

A band structure sandwiched between the solidified shells is usually observed in the cross-sectional structure of the HISTRC alloy strips with a large freezing temperature range.16–18,21,22 The central band normally includes a large amount of globular grains. The central band was also observed in the present Al–Mn–Si alloys, as shown in Fig. 3 and 4. The globular grains in the central band can be created by two different mechanisms.18 The first is attributed to the floating crystals, which nucleated in the melt pool or on the roll surface immediately after pouring. These crystals grow to be globular crystals and they are trapped in the solidifying shells from the both roll surface. Some survived globular crystals are finally sandwiched between the solid growth fronts at the roll gap to form the central band. The second is the broken twiggy dendrite branches of the solidified shell. The fragmented dendrite branches at the location near the minimum roll gap also can grow to the globular grains in the central band.
It is difficult to classify these two types from their size and morphology by the conventional optical micrographic method. However, we can distinguish them into two types, Type-I and Type-II, with the help of Weck’s reagent (Fig. 5(b)). From the SEM compositional analysis in section 3.2, and microstructure change after heat treatment in section 3.6, the presence or absence of the core structure is related to the distribution of the Si composition. The presence of the core structure represents a region having a low Si composition in the central part of the Type-I globular grains. It is considered that Si composition in the solid grown from the molten metal immediately after pouring or formed on the roll surface is low. The globular grains grown from a such solid should have the low Si region in the core. It is reasonable that only Type-I globular grains with a core structure were observed in the solidified shell at the sub-surface region. In contrast, the globular grains with no core structure, Type-II, show no large difference Si content in the grain. This suggests that they possibly grew from the molten metal with a higher Si concentration at the final solidification stage in the central band. This is why there was no Type-II globular grains in the mid-thickness solidifying shells and both the grains with/without the core-like structure existed in the central band.

These results support the formation behavior of the central band including globular grains proposed in the previous studies.17,18) The present color metallography by using Weck’s reagent is useful to judge the appropriateness of the proposed solidification mechanism of the high-speed twin-roll cast strip.

4. Conclusions

The microstructure of Al–Mn–Si alloy strip fabricated by HSTRC was characterized by color metallography using Weck’s reagent. The results of this study are summarized as follows:

(1) The microstructure of the cross section of the strip consisted of two main components: the solidified shell in the surface region and the central band in the mid-thickness region. The solidified shell was composed of cellular and equiaxed dendrites, while the central band had a large amount of globular grains. A colorful microstructure was observed by Week’s etching and a doughnut-like or necklace-like patterns (core structure) were visualized inside some globular grains. They were named Type-I globular grains for comparison of the other type with no core structure (Type-II).

(2) Quantitative observation of the tomography of etched samples and the cross-section of the film revealed that the color contrast visualized by etching resulted from the differences in the roughness of an Al substrate/ manganese oxide films’ interface, and the film thickness. The change of film’s properties was attributed to the concentration difference in Si between the central and the outer area of the grains.

(3) The compositional analysis by SEM for Type-I globular grains revealed a strong correlation between the color obtained by Week’s etching and Si distribution. The heat-treatment was applied to a cast sample to investigate this relationship. The precipitation occurred on the whole area in the Type-II grains, whereas no precipitation took place in the peripheral area of the Type-I grains. The strong relationship among Si distribution, the roughness of etched surface, characteristics of the surface film and the color revealed by the etching were confirmed.

(4) With the help of Week’s reagent, the globular grains located in the central could be classified into Type-I and Type-II based on the presence of the core structure. Type-I grains were observed in both central band and the solidified shell at sub-surface region. It is considered that the origin of Type-I grains is the floating crystals, which nucleated in the melt pool or on the roll surface. A part of the crystals is trapped in the solidifying shells from the both roll surface, and the other crystals are sandwiched between the solid growth fronts at the roll gap to form the central band. Type-II grains were observed only in the central band. They are considered to be originated from the broken twiggy dendrite branches at the location near the minimum roll gap. They grow to globular gains in the final solidification region, i.e., in the central band.

REFERENCES