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Edited by John F. Grandfield and Dmitry G. Eskin.

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FIR TREE STRUCTURE OF 1000- AND 5000-SERIES ALUMINUM ALLOY SHEET INGOTS

T. Otani
Aluminum Technical Laboratory
Showa Aluminum Industries K.K.

T. Kato
Aluminum Technical Laboratory
Showa Aluminum Industries K.K.

K. Arai
Showa Aluminium K.K.

R. Otsuka
Showa Aluminium K.K.

In many cases, a jagged, fir-tree like pattern appears on the surface of 1000- and 5000-series aluminum alloy sheets when they are anodized. This pattern has its origin in the "fir tree structure" formed in the sheet ingots.

Experiments were conducted to develop a method of preventing formation of the fir tree structure in unidirectionally solidified cast alloy. It was found that formation of the structure could be avoided if calcium is added to the cast alloy in an amount not exceeding 0.03%, preferably 0.001 to 0.008%, and the grain size is reduced to 100 μ maximum.

Use of this method in casting 7"ø to 12"ø billets and 300 x 500-mm sheet ingots of 1100, 1050 and 5005 alloys confirmed that it prevents formation of the fir tree structure in cast ingots, regardless of the size and the composition.

Identification of the intermetallic compounds crystallized in 1100 and 5005 cast alloys revealed that $FeAl_6$ had been converted to $FeAl_3$ in 1100 and $FeAl_6$ and $FeAl_m$ to $FeAl_3$ in 5005, thereby preventing formation of the fir tree structure.

1. Introduction

Formation of the fir tree structure is likely to take place in 1000 and 5000-series aluminum alloy sheet ingots. The differences in chemical properties between the outer and inner regions of the fir tree structure cause the jagged pattern to appear when the ingot is anodized after being rolled into sheet, thereby lowering the yield of on-spec aluminum sheets.

Formation of the fir tree structure is attributable to the dif ference between the Al-Fe intermetallic compound crystallized near the cast surface and those crystallized near the center of the sheet ingot. It has been reported that the composition of the crystallized intermetallic compound is affected by the sheet ingot cooling rate (1),(2) and the impurities (3) the ingot contains. Control of these factors alone, however, will not prevent formation of the fir tree structure.

Sheet ingot having the fir tree structure is used either after cutting off the edges of the structure together with parts beyond them or as is in applications where the presence of the jagged pattern in the sheet would not present any problems.

As a result of studies we made on the effect that trace elements have on the formation of the fir tree structure, we found that addition of calcium to the sheet ingot prevents formation of the structure.

Fir tree structure

Photos 1 and 2 show the fir tree structure in 1100 and 5005 aluminum alloy sheet ingots.

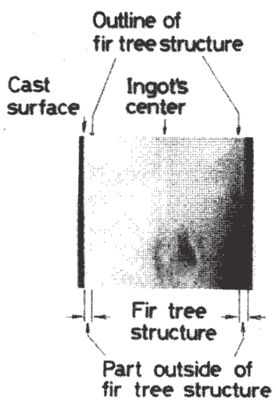


Photo 1

Fir tree structure in 1100 sheet ingot

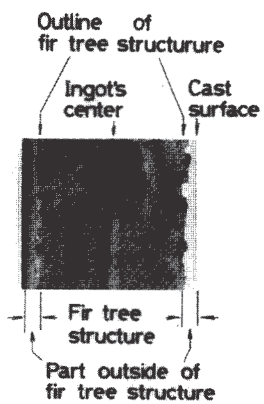


Photo 2

Fir tree structure in 5005 sheet ingot

As can be seen from the photos, the structure is closer to the center in the 5005 aluminum alloy sheet ingot than it is in the 1100 sheet ingot. The magnesium contained in the 5005 sheet ingot is said to cause such difference (3). $FeAl_6$ crystallizes in the fir tree structure and remains in the anodic film without being oxidized by anodization, thereby causing the anodic film to take on a dark gray color. On the other hand, $FeAl_3$ and $FeAl_m$ that crystallize outside the structure are oxidized by anodization and do not remain in the anodic film, thereby causing the anodic film to assume a light gray color.

Fig. 1 shows the results of the measurement made of the dendrite arm spacing (hereinafter referred to as "DAS") to infer the cooling rate in the 1100 alloy sheet ingot.

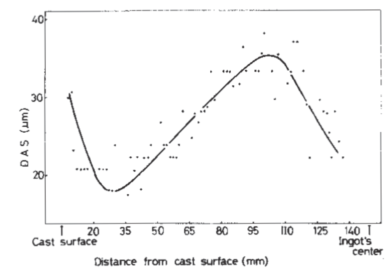


Fig. 1 DAS of 1100 sheet ingot

The DAS reaches its minimum about 20 to 35 mm from the cast surface, becomes larger at points closer to the center of the ingot and decreases again in the central area. Although the sheet ingots have almost the same DAS curve under the same solidifying conditions, the outline of the fir tree structure appears at different spots in the sheet ingot: in one case, close to the cast surface and, in the other, about 60 mm from it.

Although it is common practice among aluminum rolling companies to diminish the effect of the fir tree structure by employ ing the lowest possible casting speed and thereby cause the structure to extend as close to the cast surface as possible, there is no conventional method of eliminating it. In the casting test explained immediately below, however, the fir structure is caused to shrink crosswise until it disappears.

3. Test method

3.1. Unidirectional solidification test

A 2-kg piece of aluminum alloy was melted in a silicon carbide crucible and cast in a 30ø x 150-mm mold. While the outside surface of the mold was heated to 600°C, the bottom of the cast alloy was cooled direct with water at the same cooling rate as in the case of the sheet ingot. The cast alloy was then cut longitudinally into two pieces and after anodizing one of them for 50 minutes with the current set at 2.5 A/dm^2, the distance from the bottom surface of the cast alloy to the edge of the fir tree structure in it was measured and defined as fir tree height.

3.2. Continuous casting test

With the results of the unidirectional solidification test used as basic information, 300 kg of aluminum alloy was melted to continuously cast 6 to 12"ø billets and 300 x 500-mm sheet ingots for the purpose of observing the growth of the fir tree structure.

4. Test results

4.1. Results of unidirectional solidification test

4.1.1. Comparison of unidirectionally solidified cast alloy with continuously cast sheet ingot.

1100 alloy was melted and 0.014% of titanium was added in the form of Al-5%Ti-1%B in producing unidirectionally solidified cast alloy. Pho to 3 shows the typical jagged pattern that appeared on it upon anodization.

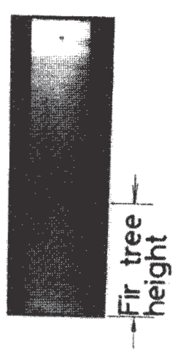


Photo 3 Jagged pattern on unidirectionally solidified alloy

It is very similar in appearance to the jagged pattern formed on the sheet ingot.

Fig. 2 shows the DAS of the unidirectionally solidified cast alloy. The DAS ranges from 10 to 30 μm, indicating a similarity to the DAS of the sheet ingot shown in Fig. 1.

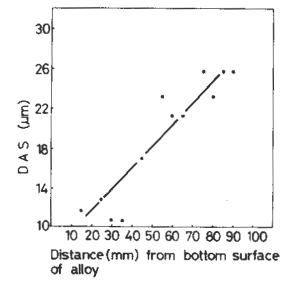


Fig. 2 DAS of unidirectionally solidified alloy

It was judged from the above fact that unidirectionally solidified cast alloy could be used in simulation of sheet ingot.

4.1.2. Grain size and fir tree height

Fig. 3 shows the relationship between fir tree height and grain size in the case of 1100 alloy, the grains of which had been reduced to very small particles by the addition of titanium in the form of Al-5%Ti-1%B.

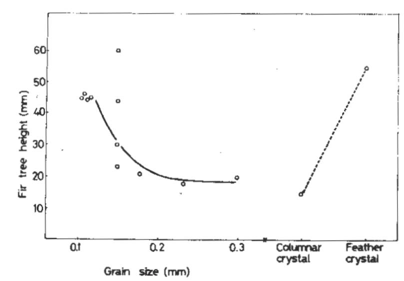


Fig. 3 Fir tree height vs. grain size

The larger the grains, the lower the height, the maximum height being 20 mm when the grain size is 300μ. Judging from this phenomenon, it is conceivable that enlarging of the grains by reducing the amount of the grain refiner will enable the height to be lowered to the extent required to eliminate the fir tree structure from the sheet ingot.

Enlargement of the grains by such means, however, will facilitate the growth of feather crystals, which will, as indicated in Fig. 3, cause the structure to be formed at a high level as in the case where the grains are smaller.

4.1.3. Addition of trace elements and fir tree structure

A study was made of the effect of lithium, beryllium, potassium, barium and calcium on formation of the fir tree structure, when added to the 1100 alloy. The results are given in Fig. 4.

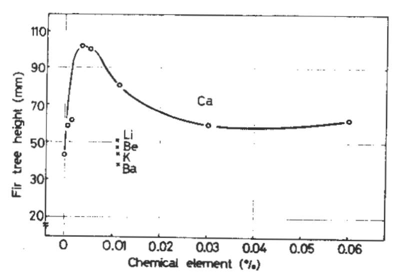


Fig. 4 Chemical elements vs. fir tree height

Although the fir tree height was practically the same when lithium, beryllium, potassium or barium was added in the amount of 0.01%, it markedly increased when calcium was added in the same amount. The amount of calcium was then changed to clarify the effect, and it was found that an addition of about 0.005% maximized the height and that of 0.03% or more did not bring about any substantial change in height in comparison with the case of no addition.

4.1.4. Interaction between calcium and grain refiner

Fig. 5. shows the change in fir tree height when the grain refiner in the form of Al-5%Ti-1%B or Al-5%Ti was added, with the amount of calcium fixed at 0.003%.

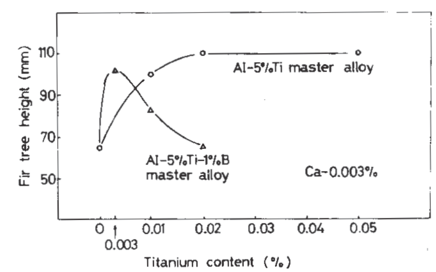


Fig. 5 Interaction between calcium and grain refiner

When A1-5%Ti-1%B was used, the height reached at its maximum with addition of 0.003% titanium and went down with increased additions. In the case of A1-5%Ti, however, the height reached its maximum when 0.02% titanium was added and did not change with increases in the amount. Although the maximum height reached by the fir tree was 60 mm, as shown in Fig. 3, when calcium was not added, it rose close to 110 mm when calcium was added.

Judging from this, it is conceivable that addition in proper quantities of calcium and the grain refiner will enable elimination of the fir tree structure.

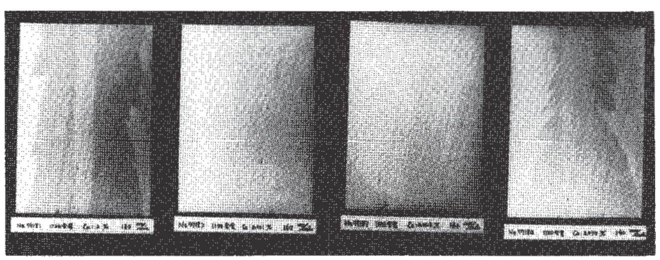
4.2. Results of continuous casting test

A continuous casting test was conducted to confirm the capability of calcium to eliminate the fir tree structure, as had been demonstrated in the unidirectional solidification test.

First, 0.01% titanium in the form of A1-5%Ti-1%B was added to molten 1100 alloy, and four 7"ϕ billets were cast from the alloy at a rate of 160 mm/min, the first one without addition of calcium; the second after adding 0.001% calcium; the third, 0.008% and the last, 0.030%. Photo 4 shows the anodized longitudinal sections of the billets.

Composition of 1100 alloy

Fe	Si	Cu	Ti	Al
0.50	0.10	0.10	0.01	Bal



Ca-0%　　Ca-0.001%　　Ca-0.008%　　Ca-0.03%

Photo 4 Anodized longitudinal sections of billets cast from 1100 alloys with calcium and without calcium

As can be seen in the photo, the jagged pattern appeared on the first billet, but not on the second and the third, but appeared on the last one. Similar results were obtained in the unidirectional solidification test. Although the billet diameter was enlarged to 8, 10 and 12 inches, the results were the same. Lowering of the casting speed to 100 mm/min did not affect the results, the only exception being that the fir tree structure in the first and the last billet drew closer to the cast surface than in the case of casting at 160 mm/min.

Knowing that no fir tree structure was formed in the 1100 alloy containing 0.01% titanium as well as 0.001 or 0.008% calcium, we cast a 300 x 500-mm sheet ingot from 1100 alloy to which the same amount of titanium and 0.003% calcium had been added. No growth of the structure was observed in the sheet ingot as well. The same result was obtained with 1050 and 5005 alloy sheet ingots of the same size.

Photos 5-1 and 5-2, which show longitudinal sections of a 10"ϕ billet, were taken to confirm the necessity of adding not only calcium but also the grain refiner to eliminate the fir tree structure. Photo 5-1 shows the jagged pattern that appeared on the surface of an andized section and Photo 5-2, the macrostructure observed on the surface of a non-anodized section.

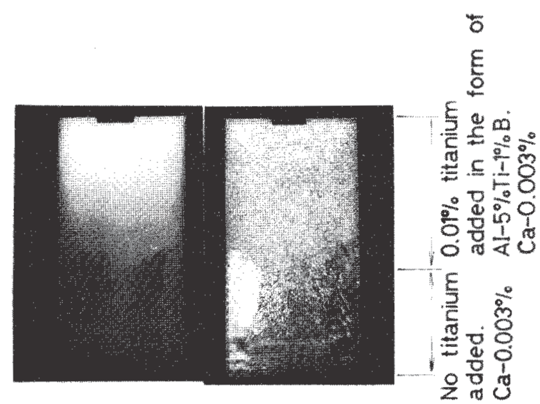


Photo 5-1　　Photo 5-2

Since no titanium was added to the lower part of the billet, a feather crystal and large grains were formed there, as can be seen in Photo 5-2, and, when anodized, the jagged pattern appeared, as shown in Photo 5-1. In the upper part of the billet, however, 0.01% titanium in the form of Al-5%Ti-1%B was added, causing the fir tree structure to shrink crosswise until it disappears, as seen in Photo 5-1.

4-3. Advantages of new method

The new method has the following advantages:

1) There is little difference in color tone from one lot of anodized products to another.

2) The cast alloy yield improves, because the fir tree structure is not formed, even at the bottom of the casting.

3) The casting speed is faster.

4) Formation of the fir tree structure can be easily prevented even in 1050 and 5005 alloys, in which it is easy for the structure to grow.

5. Observations

5.1. Interaction between calcium and grain refiner

The results of the unidirectional solidification test given in Fig. 5, make it clear that the effect of calcium on the fir tree height varies with the kind and the quantity of the grain refiner. Fig. 6-1 shows the grain sizes of the alloys used in the unidirectional solidification test. When Al-5%Ti-1%B was added, the grain size was reduced to below 100μ with only 0.003% titanium and, in the case of Al-5%Ti, the same result was obtained with 0.02% titanium. In other words, the amount of titanium enabling the fir tree height to reach its maximum in each case as seen in Fig. 5 agrees with that which reduces the grain size to below 100μ, as seen in Fig. 6-1.

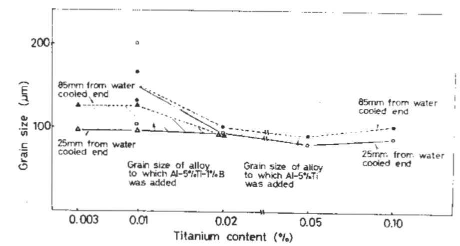


Fig. 6-1 Grain sizes of unidirectionally solidified alloys

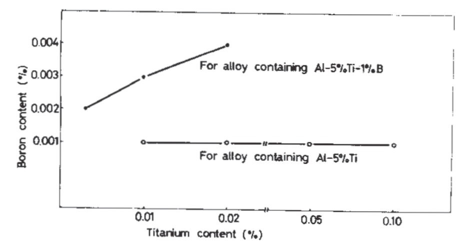


Fig. 6-2 Titanium content vs. boron content

With the addition of 0.003% titanium in the form of Al-5%Ti-1%B, the fir tree height reaches its maximum and as the amount of the additive increases, the height becomes lower. This phenomenon was considered as having something to do with the amount of boron mixed together with the titanium into the alloy. The relationship between the amount of boron and that of titanium in 1100 alloy as given in Fig. 6-2 indicates that the amount of boron increases with an increase of that of titanium added in the form of Al-5%Ti-1%B.

Photos 6-1, 6-2 and 6-3 were taken through an EPMA.

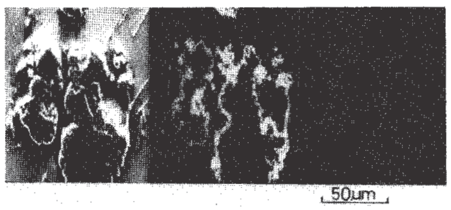


Photo 6-1 Photo 6-2 Photo 6-3

Photo 6-1 shows $A1B_2$ particles contained in 1100 alloy to which boron and calcium had been added in the amount of 0.1% each; Photo 6-2, calcium gathered around the particles and Photo 6-3, boron present in the particles. Since boron combines with calcium to form a chemical compound, a part of the calcium added to the alloy is considered to unite with boron mixed in the form of A1-5%Ti-1%B, and as the amount of boron increases with an increase of the amount of titanium caused by adding the master alloy, the amount of calcium that is effective for elimination of the fir tree structure is considered to decrease by its combination with the boron, thereby lowering the fir tree height.

5-2. Identification of A1-Fe intermetallic compounds

Al-Fe intermetallic compounds formed in and out of the fir tree structure in 1100 and 5005 sheet ingots were analyzed and are schematically presented (4) in Fig. 7.

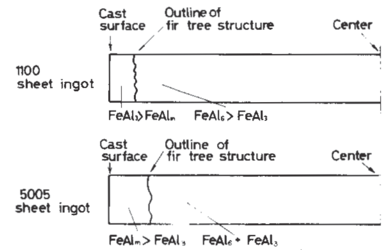


Fig. 7 X-ray identification of compounds

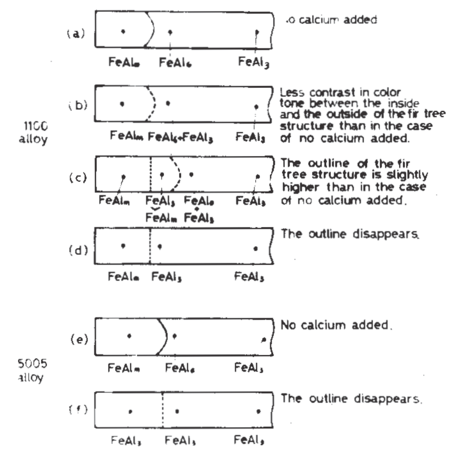


Fig. 8 Identification of A1-Fe intermetallic compounds in unidirectionally solidified cast alloy

Figs. 8(a) to (f) show the results of identification of crystallized Al-Fe intermetallic compounds in unidirectionally solidified 1100 and 5005 alloys. Calcium was added to all the alloys except those represented by Figs. 8(a) and (e). Figs. 8(d) and (f) indicate that, with addition of the proper amount of calcium, the $FeAl_6$ phase is replaced by the $FeAl_3$ phase in the 1100 alloy to remove the fir tree structure, and also $FeAl_6$ and $FeAl_m$ are converted to $FeAl_3$ in the 5005 alloy to remove the structure. Although change of the $FeAl_6$ phase to the $FeAl_3$ phase is referred to in a report (1) on the cooling rate of the alloys, and the composition in a report (2) on the effect of Fe-to-Si ratio, the amount of magnesium, etc. on the location of the fir tree structure, no report has been published on the effect that addition of a minute amount of a third element, such as calcium, has on its location. In order to examine the effect of calcium, therefore, we checked, using the EPMA, the distribution of calcium in Al-Fe intermetallic compounds that had been extracted from 1100 and 5005 alloys. As a result, many calcium particles were detected in the intermetallic compounds, but none in the case matrix. A number of titanium particles were also seen in them.

Judging from this and the results of identification of intermetallic compounds in 1100 and 5005 alloys, it is presumed that calcium and titanium facilitate cystallization of Al-Fe intermetallic compounds. Further study is being made for the purpose of verification.

6. Conclusion

Tests were conducted to determine the effect of lithium, beryllium, potassium, barium and calcium on the formation of the fir tree structure in 1000-series aluminum alloy and of that in 5000-series alloy, when they were added in the amount of 0.01% separately to the alloys in a molten state. It was found that addition of calcium can cause the fir tree structure to shrink crosswise until it disappears, if the grain size can be kept at 100 maximum and the amount of the calcium is not more than 0.03%, preferably, 0.001 to 0.008%. Also, identification of the Al-Fe intermetallic compounds in the alloys from which the fir tree structure was eliminated, revealed that the $FeAl_3$ phase had replaced the $FeAl_6$ phase in the 1100 alloy, and $FeAl_m$ and $FeAl_6$ phases in the 5005 alloy.

References:

(1) L. Baecherud, Jernkont. Ann., Vol 152, p 109, 1968

(2) Kosuge & Mizukami, Journal of the Japan Institute of Light Metals, Vol 25, p 48, 1975

(3) Asami, Nishitsuji & Tanaka, Proceedings of the 53rd Autumn General Meeting of the Japan Institute of Light Metals, p 11

(4) Asami, Tanaka & Hideno, Journal of the Japan Institute of Light Metals, Vol 28, p 321, 1978