IMPROVEMENT OF HOMOGENEITY IN LARGE DIAMETER C.P. TITANIUM INGOTS
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Introduction
In pursuit of higher yield in manufacturing welded heat exchanger tubes for power and chemical plants, mill product manufacturers (our customers), increasingly enlarging the width of cold strips, are demanding large diameter ingots with very narrow range of specification of chemical components, particularly iron and oxygen, over the entire ingot so as to minimize fluctuation in mechanical properties of strips.

Since 1981 when our new melting plant was built, we have been producing large diameter double melted C.P. titanium ingots, approximately 1 meter in diameter and 7-8 tons in weight, by using a consumable electrode vacuum arc melting furnace. At the start operation, it was thought difficult to obtain homogeneous large diameter ingots because of segregation due to solidification. Since few have reported on this problem, experiments were conducted with the aim of improving the homogeneity of large diameter ingots.

This report concerns the homogeneity of large diameter C.P. titanium ingots and discusses experiments on the effects of secondary melting conditions, particularly in the later part of the melting, and of adjustment made in the chemical components of a primary electrode.

Test methods
Fig. 1 shows the outline of our production process of C.P. titanium ingots.

![Diagram of production process](image)
It is important to reduce the segregation of chemical components in the top portion of the secondary ingot in order to obtain large diameter ingots of better homogeneity. Therefore, tests were performed on four different melting current patterns with various hot topping times in secondary melting. In addition, the primary electrode was manufactured so that iron and oxygen contents in its lower 15% portion, (which becomes the top of the secondary ingot) were 100 ppm lower and 200 ppm higher respectively than the target values.

The test ingots were C.P. titanium ingots whose chemical component target values are shown in Table 1.

<table>
<thead>
<tr>
<th></th>
<th>Fe</th>
<th>O</th>
<th>N</th>
<th>C</th>
<th>H</th>
</tr>
</thead>
<tbody>
<tr>
<td>Target values (ppm)</td>
<td>500</td>
<td>1300</td>
<td>&lt;100</td>
<td>&lt;100</td>
<td>&lt;50</td>
</tr>
</tbody>
</table>

The secondary ingots were cut longitudinally to examine the segregation of chemical components in detail as well as to observe the macrostructures in the longitudinal sections.

Test results
(1) Segregation of chemical components
Fig. 2 shows the secondary melting current patterns and the distribution of iron and oxygen in the ingot longitudinal section.

For melting current pattern A, with extremely short hot topping time, iron and oxygen contents deviate most from the target values at a point about 500 mm below the top; iron shows positive segregation (+200 to +300 ppm) whereas oxygen shows negative segregation (lower than -300 ppm). Though the ingot top portion near the surface partly comes close to target values, most of the top portion shows large segregation. (iron: +100 to +200 ppm; oxygen: -200 to -300 ppm)

For pattern B, with a relatively short hot topping time, segregation is less than in pattern A, though it is still considerably large. In contrast, for patterns C and D, with relatively long hot topping time, segregation is remarkably reduced, resulting in deviation from the target values within the range -100 to +100 ppm for iron and +100 to -200 ppm for oxygen.

Pattern E, with chemical composition adjusted when the primary electrode is manufactured, shows a distribution similar to that for pattern C, though the hot topping time is the same as for pattern B.

For all patterns A to E, the middle and bottom portions of the
Fig. 2-1: Melting current patterns and iron in ingot longitudinal section.
### Fig. 2-2: Melting current patterns and oxygen in ingot longitudinal section.

<table>
<thead>
<tr>
<th>Melting current (kA)</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time (min.)</td>
<td>4</td>
<td>30</td>
<td>100</td>
<td>170</td>
<td>25</td>
</tr>
<tr>
<td>Deviation from target (ppm)</td>
<td>+100~100</td>
<td>-100~200</td>
<td>-200~300</td>
<td>-300~400</td>
<td>Not analyzed</td>
</tr>
</tbody>
</table>

**Distribution of oxygen**

- **A**

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**Secondary melting current pattern**

- **A**
- **B**
- **C**
- **D**
- **E**

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- **Adjustment (Top)**
  - **A**

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- **Adjustment (Bottom)**
  - **A**

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**Note:** The diagram illustrates the melting current patterns and the distribution of oxygen in an ingot's longitudinal section, showing the deviation from the target value in parts per million (ppm).
ingots are homogeneous, with less segregation compared with the top portion.

Fig. 3 shows the relationship between hot topping time in the secondary melting and the maximum deviation from target values for chemical components.

For both iron and oxygen the deviation decreases as the hot topping time increases. In patterns C and D, for example, deviation from the target values is not more than 70 ppm for iron and 140 ppm for oxygen. In actual operation, however, the melting time should be as short as possible, in view of productivity. Therefore, we determine the actual hot topping time and chemical composition adjustment for the primary electrode so as to satisfy both productivity and customer specifications, and establish the melting standard on that basis.

Fig. 4 shows the diametrical distribution of iron and oxygen in detail.

Line A-A corresponds to line A-A in Fig. 2 and is considered to indicate the final solidified position. For pattern A, both iron and oxygen show very large segregation in the center portion of the ingot top. For patterns C and D, the segregations of iron and oxygen in the center are substantially reduced compared with those in pattern A. Specifically, the maximum degree of iron segregation is reduced from 1.44 for pattern A to 1.14 for patterns C and D, and that of oxygen from 0.74 to 0.89. In the middle and bottom portions of the ingot as well, segregation due to solidification can be observed in the center, though it is not as conspicuous as in the top portion.

For pattern E, segregation is reduced to almost the same level as for patterns C and D, with longer hot topping time.

(2) Macrostructures

Photograph 1 shows an example of the macrostructure in a longitudinal section of the ingots.

As shown, solidification mainly proceeds upwardly from the bottom in the bottom portion, leaving the flat-bottomed molten pool. In the middle portion, columnar crystals are formed about 150-300 mm from the surface, growing from the ingot mold wall side. Equiaxed crystals are observed in the center portion of the ingot. According to V.V. Tetyukhin, et al. (1), the thickness of the columnar crystals varies in direct proportion to current density, becoming narrower with a wider solidifying temperature range.

The final solidified position is about 500 mm from the ingot top for pattern A and about 300 mm from the top for patterns C and D. Thus, influence of the secondary melting conditions can be clearly seen in macrostructures as well, the solidification pattern coinciding with the segregation pattern of chemical components.
Fig. 3: Effect of hot topping time on chemical component deviation.

Oxygen
Iron
Adjustment of iron and oxygen in top portion

Hot topping time (min)

Fig. 4: Segregation of iron and oxygen in diametrical direction of ingot

Photo 1: Macrostructure in longitudinal section of ingot melted with pattern D.
Discussion
Since it is assumed that segregation of the chemical components of an ingot is due to solidification, the depth of the molten pool during the secondary melting is important. The molten pool is basically parabolic in shape. V.I. Dobatkin, et al. (2-3) have formulated an equation (1) for calculating molten pool depth as follows:

$$H = 5(1 - 0.25D)V$$

Equ. (1)

where

$H$: Molten pool depth (m)
$D$: Diameter of ingot (m)
$V$: Melting rate (kg/sec)

Table 2 shows the molten pool depth values calculated using Equation (1) and those estimated from the macrostructures, both under the secondary melting conditions for this test.

Table 2

<table>
<thead>
<tr>
<th>Melting current</th>
<th>Melting rate</th>
<th>Diameter of ingot</th>
<th>Molten pool depth $H$ (m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$I$ (kA)</td>
<td>$V$ (kg/sec)</td>
<td>$D$ (m)</td>
<td>Calculated</td>
</tr>
<tr>
<td>36</td>
<td>0.40</td>
<td>0.98</td>
<td>1.51</td>
</tr>
<tr>
<td>18</td>
<td>0.24</td>
<td>0.98</td>
<td>0.91</td>
</tr>
<tr>
<td>16</td>
<td>0.21</td>
<td>0.98</td>
<td>0.79</td>
</tr>
</tbody>
</table>

As shown, the calculated values are considerably higher than the estimated values. This is probably because of the cooling effect from the bottom. It is easily inferred from these calculated values and the estimated values that qualitatively, the molten pool depth varies widely, depending upon the melting current. Therefore, the shape of the molten pool is assumed to vary depending upon the hot topping time of the secondary melting, as illustrated in Fig. 5.

Fig. 5: Schematic diagram of molten pool at end of melting

$x$: Final solidified position
$y$: Molten pool depth at end of melting
$H$: Molten pool depth at starting of hot topping.
For pattern A, the molten pool at the end of melting is considerably deep as is the case with stable melting current. A deep molten pool results in large heat capacity and latent heat of solidification, which in turn retards solidification in the center portion. At the final solidified position, therefore, iron with an equilibrium distribution coefficient of less than 1 presents positive segregation and oxygen with a coefficient larger than 1 presents negative segregation. For patterns C and D, in contrast, molten pool depth gradually reduces until it reaches a fairly small value at the end of melting. As a result, the heat capacity and latent heat of solidification are sufficiently small to permit fairly uniform solidification, thus reducing the segregation of chemical components.

As discussed above, segregation of the chemical components in an ingot can be qualitatively explained by use of solidification patterns and equilibrium phase diagrams for Ti-Fe and Ti-O systems.

Conclusion

We studied the homogeneity of large diameter double melted C.P. titanium ingots produced by a consumable electrode vacuum arc melting furnace, and obtained various data on segregation due to solidification.

The segregation of the chemical components in an ingot can be qualitatively explained by the solidification patterns and equilibrium phase diagram. To reduce segregation, it is necessary to control hot topping conditions in the later part of the secondary melting so as to gradually reduce the molten pool and achieve fairly uniform solidification.

Since reduction in hot topping time is essential to better productivity, the secondary melting current pattern is determined according to customer specifications. A technique of adjusting the chemical composition of the primary electrode in the longitudinal direction has been also put into practical use so as to satisfy both productivity and homogeneity simultaneously.

Thus, we are producing and supplying homogeneous ingots whose chemical components, particularly iron and oxygen, are kept within a narrow range so as to meet customer needs. We trust that we are contributing to the improved homogeneity of mechanical properties and grain size of mill products.

References