METHOD FOR THE PRODUCTION OF FINE GRAINED STEEL

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ABSTRACT OF THE DISCLOSURE

Method of producing fine grained steel by adding to said steel prior to teeming an alloy containing aluminum and silicon and another alloy containing vanadium and silicon. This invention relates to a novel method for treating molten steel to produce fine grain size in the finished product. When carbon or alloy steel is heated above its transformation temperature, it enters into a solid solution phase known as austenite. Austenite is a crystalline structure and the size of the austenite crystals or grains is a function of the degree to which the steel is heated beyond the transformation temperature. As the temperature increases beyond the transformation temperature, the austenite grain size also increases. The grain size of the cooled steel is determined by the austenite grain size so that it is readily apparent that the final grain size will be determined by the maximum temperature to which the steel is heated. The grain size of the final product has a great effect upon the properties of the product. Since heat-treatment processes, such as hardening, normalizing and annealing require that the steel be heated to temperatures in excess of the transformation temperature, it is necessary to inhibit austenitic grain growth where a fine grain size is desired. For example, carburizing steel for case hardening is most commonly performed at about 1700° F. which is well above the transformation temperature of the austenitic phase. Such steels must remain fine grained after treatment.

Steels with large grains are termed coarse grained, while steels with small grains are termed fine grained. The McQuaid-Ehn test is commonly used for determining the austenitic grain size, and the ASTM grain size numbers are normally used for rating the steels. An ASTM rating of 1 to 5 is considered coarse, while an ASTM rating of 5 to 8 is considered fine. Grain size ratings finer than 8 may also occur. It is to be understood that the term fine grained steel, when used hereinafter, refers to steel having an ASTM number of 5 to 8 and finer as determined by the McQuaid-Ehn grain size test, unless otherwise specified.

Fine grained heat treated steels are normally preferred over coarse grained steels because, for a given hardness, the fine grained steel is tougher, residual stresses are lower, there is less distortion, quenching cracks are less prevalent and the steel has less retained austenite. For this reason, melting practices are used which will produce a fine grained steel. The practices include additions prior to and during tapping which tend to inhibit austenitic grain growth in the final product.

Aluminum is most commonly used to produce fine grained steels because it is both effective and inexpensive. Normal aluminum additions for producing a fine grained plain carbon steel range from 1 lb./ton of steel to 5 lb./ton of steel depending upon the grade of steel and the steel-making practice. A residual aluminum content of .02 to .05% is desired to produce a fine grain structure. With alloy steels the residual aluminum may be as low as .015% to produce a fine grain structure.

However, there are many cases where the addition of aluminum to the steel is detrimental. For instance transverse mechanical properties of plate products from such steels are lower than longitudinal mechanical properties, particularly in impact resistance. Another detrimental effect of aluminum is that it creates a poor surface on the ingot or wrought product and this results in costly conditioning, poor formability or even rejection. Furthermore, aluminum cannot be added to some grades of steel because it causes poor hot-workability. Still another detrimental effect of aluminum has recently come to light with the advent of continuous casting. In that the addition of sufficient aluminum to the steel in the ladle to produce a fine grained steel causes the molten metal to solidify in the relatively small bushing nozzles.

It is known that the addition of other elements, such as vanadium, columbium, titanium, zirconium, either singly or in combination, will produce fine grained steels. Apart from aluminum the element most commonly used for this purpose is vanadium. The amount of vanadium normally added, as ferrovanadium, is .08% to .15%. While the use of vanadium is effective, it is relatively expensive, costing approximately ten times more per pound than aluminum.

My novel method is directed to the treatment of molten carbon and alloy steels to produce a high quality, fine grained steel in the cast or wrought condition. The steel treated in accordance with my method may be continuously cast and retains its fine grain structure at high temperatures such as are encountered in high temperature carburizing. Additionally, my method is inexpensive since it requires a very small amount of vanadium.

I have discovered that the addition of a small amount of aluminum, insufficient by itself to produce fine grained steel, plus the addition of a much smaller amount of vanadium than that required to produce fine grained steel when used alone in accordance with previous practices, will produce a fine grained steel. In my novel method it is essential that the aluminum be added as an alloy containing both aluminum and silicon rather than as aluminum metal. Additionally, it is essential in my method that the vanadium be added as an alloy containing vanadium and silicon rather than as ferrovanadium. The alloys disclosed in my copending application Ser. No. 502,380, filed on even date herewith are especially adapted for this purpose.

I have also discovered that if the alloy of vanadium and silicon contains small amounts of one or more carbide and nitride forming elements, for example, columbium, titanium, zirconium and boron, even smaller additions of vanadium produce a fine grained steel. When an alloy including carbide and nitride forming elements is used, the steel remains fine grained after heating to temperatures in excess of 1700° F. A vanadium-iron-silicon alloy including some of the elements specified above is termed complex V-Fe-Si and is disclosed in my copending application Ser. No. 502,380.

Basically my method consists in treating molten steel prior to teeming by adding an aluminum-iron-silicon alloy and a vanadium-iron-silicon alloy to the molten metal. The addition of aluminum and vanadium in the form of silicon alloys as distinguished from aluminum metal and ferrovanadium produces a fine grained steel which may be treated at temperatures well within the austenite range while maintaining a fine grain size.

In order to determine the grain size of steel produced by my process, AISI 1040 steel heats were melted in a 300 pound induction furnace and various additions were made to the molten metal after it was tapped into a ladle. The ladles of steel were then teemed into 4" x 4" x 24" molds. The resulting ingots were identified and forged to 1" x 2" square bars. Sections were cut from these bars for
the McQuaid-Ehn grain size tests and the ASTM grain size was determined after heat treatment.

In melting the steel heats, Armco scrap was melted in the induction furnace, the slag removed and sufficient silicon added as 50% FeSi to lower the FeO content and leave a residual of 0.10% silicon in the bath. Sufficient pig iron was added to give .40% carbon and when in solution, electrolytic manganese and ferrosilicon were added to produce the required composition. The heats were then tapped at 2900° F. into the ladle and treated.

The analysis of the steels after treatment met the AISI 1040 specification and had the following composition ranges:

<table>
<thead>
<tr>
<th>Element</th>
<th>Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>C</td>
<td>0.41-0.43</td>
</tr>
<tr>
<td>Mn</td>
<td>0.82-0.88</td>
</tr>
<tr>
<td>P</td>
<td>0.010-0.012</td>
</tr>
<tr>
<td>S</td>
<td>0.017-0.019</td>
</tr>
<tr>
<td>Si</td>
<td>0.24-0.32</td>
</tr>
</tbody>
</table>

The treatment in the ladle in each instance was to deoxidize with 2 lbs. of calcium-ferrosilicon alloy per ton of steel when the ladle was half full. This addition was followed by .02% aluminum addition as either aluminum or an aluminum-ferrosilicon alloy. This was in turn followed by the addition of vanadium as either ferrovanadium or a vanadium-iron-silicon alloy.

The various ladle additions had the following compositions:

- **Calcium-ferrosilicon alloy**
  - 16.0% Ca, 57.8% Si, bal. essentially Fe.

- **Aluminum-iron alloy**
  - 19.0% Al, 38.9% Si, bal. essentially Fe.

- **Ferrovanadium**
  - 75.15% V, bal. essentially Fe.

- **V-Fe-Si (Ht. 15)**
  - 28.00% V, 51.48% Si, bal. essentially Fe.

- **V-Fe-Si (Ht. 28)**
  - 51.34% V, 24.82% Si, bal. essentially Fe.

- **Complex V-Fe-Si (Ht. 16)**
  - 29.55% V, 46.12% Si, 2.31% C, 2.04% Ti, 1.98% Zr, 0.11% B, 1.75% Al, 1.59% Ba, 2.34% Mn, bal. essentially Fe.

It will be observed that the addition of .02% aluminum to AISI 1040 steel, either as aluminum or as an alloy containing aluminum and silicon produced a coarse grained steel.

**EXAMPLE 2**

Additional ingots were made and processed in the manner described in Example 1. The ladle additions and the grain size results for the individual heats are listed below.

<table>
<thead>
<tr>
<th>Heat No.</th>
<th>Ladle Additions</th>
<th>McQuaid-Ehn Grain Size, 1,700° F.</th>
</tr>
</thead>
<tbody>
<tr>
<td>14A</td>
<td>.02% Al as Aluminous + .00% V as V-Fe-Si (Ht. 15)</td>
<td>7, 8 plus fine, 6°</td>
</tr>
<tr>
<td>21C</td>
<td>.02% Al as Aluminum + .04% V as Duplex 5-8 plus 1-4</td>
<td></td>
</tr>
<tr>
<td>21B</td>
<td>.02% Al as Al-Fe-Si + .04% V as Duplex 5-8 plus 1-4</td>
<td></td>
</tr>
</tbody>
</table>

With an addition of .02% aluminum the minimum vanadium addition as ferrovanadium which produced a fine grained steel was .06%. Below .06% vanadium the steels contained coarse grains of sizes No. 1 to 4 regardless of whether the aluminum was added as aluminum metal or as the aluminum-iron-silicon alloy as shown by a comparison of Heats 21C and 21B.

**EXAMPLE 3**

Additional ingots were made and processed in the manner described in Example 1. The ladle additions and the grain size results for the individual heats are listed below.

**EXAMPLE 4**

Additional ingots were made and processed in the manner described in Example 1. The ladle additions and the grain size results for the individual heats are listed below.

<table>
<thead>
<tr>
<th>Heat No.</th>
<th>Ladle Additions</th>
<th>McQuaid-Ehn Grain Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>24C</td>
<td>.02% Al as Aluminum + .04% V as Duplex 5-8 plus 1-4</td>
<td></td>
</tr>
<tr>
<td>24B</td>
<td>.02% Al as Al-Fe-Si + .04% V as 7, 8 and finer</td>
<td>V-Fe-Si (Ht. 15)</td>
</tr>
</tbody>
</table>

When the vanadium was added to the steel as the V-Fe-Si alloy, a fine grained steel was produced at the lower vanadium level but, as shown by a comparison of heats 24B and 24C, only when the aluminum was added as an alloy containing silicon. It is pointed out that the grain size for Heat 24C is the same as for Heats 21B and 21C in Example 2.

**EXAMPLE 5**

With an addition of .02% aluminum as an alloy containing silicon, extremely low additions of vanadium as the V-Fe-Si alloy or as the complex V-Fe-Si alloy containing small amounts of other elements produced fine grained steel. The steels treated with the complex V-Fe-Si alloy were also fine grained at higher temperatures. To show that the fine grain at 1800° F. is a characteristic of the complex V-Fe-Si alloy used and not the level of the vanadium addition, the higher vanadium additions of .04% as each of the two vanadium alloys containing silicon are included in the example. In all of the above tests the core of the carburized specimens had the same grain size as the grain size recorded for the case.

The addition of the calcium-ferrosilicon deoxidizer prior to the aluminum-iron-silicon alloy addition is not essential but assures more uniform aluminum recoveries. Other deoxidizers could be used if desired.
The above examples demonstrate that the aluminum must be added as an alloy of aluminum containing silicon, and that the vanadium must be added as an alloy of vanadium containing silicon. Example 4 also demonstrates that when the molten steel is treated according to my novel method using the complex vanadium alloy disclosed in my copending application, the amount of vanadium necessary to assure fine grain size is far less than the amount required when other vanadium alloys are used.

In addition it has been demonstrated that minute amounts of vanadium, when added as the novel complex vanadium silicon alloy containing small amounts of elements such as columbium, titanium, zirconium, boron, etc., and in accordance with my novel method, will produce a fine grained steel which steel will be fine grained at higher temperatures such as used for high temperature carburizing. The amounts of the effective elements other than vanadium in the .0025% vanadium addition (Hit. 30D) were extremely minute and, except for vanadium, are too low to be determined quantitatively in the finished steel. Based on the knowledge of grain size control of steel, such levels of addition, either singly or in combination, would not have any effect on the grain size of the steel.

My invention may be embodied within the scope of the appended claims.

1. The method of producing fine grained steel comprising adding to molten steel prior to teeming aluminum as an alloy containing aluminum and silicon and adding vanadium as another alloy containing vanadium and silicon and teeming said molten steel.

2. The method set forth in claim 1 wherein said aluminum addition is less than about .02%.

3. The method set forth in claim 1 wherein said vanadium addition is about .005%.

4. The method set forth in claim 1 wherein said aluminum addition is less than about .02% and said vanadium addition is about .005%.

5. The method set forth in claim 1 wherein said alloy containing vanadium and silicon includes small amounts of columbium, titanium, zirconium and boron.

6. The method set forth in claim 1 wherein said alloy containing vanadium and silicon includes small amounts of columbium, titanium, zirconium and boron, and said vanadium addition is .0025%.

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