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3,383,202
GRAIN REFINING ALLOY
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mesne assignments, to Foote Mineral Company, Exton,
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3 Claims. (Cl. 75-122)

ABSTRACT OF THE DISCLOSURE

Alloy for controlling grain size in cast or wrought steel consisting essentially of 30-60% silicon, up to 15% manganese, up to 15% calcium plus barium, a total of 20-40% of at least four elements from the group consisting of cerium, lanthanum, columbium, tantalum, vanadium, zirconium, titanium, aluminum and boron, with the balance iron and incidental impurities.

This invention relates to novel alloy for addition to steel to produce a steel having fine grain size. When carbon or alloy steel is heated above its transformation or critical temperature as in heat treatment, it enters into a solid solution phase known as austenite. Austenite is a crystalline structure and the temperature to which steel is heated beyond the transformation temperature will determine the size of the austenite crystals or grains, the grain size generally increasing as the temperature increases. The grain size in the cooled steel is in turn determined by the austenitic grain size. Since a fine grained heat treated steel is normally preferably for a given hardness, it is desirable to inhibit the austenitic grain growth. For a given hardness a fine grained steel is tougher than a coarse grained steel, has lower residual stresses, less distortion and quenching cracks are less prevalent. The usual practice in producing fine grained steel has been to add aluminum to the steel as it is tapped into the ladle. Aluminum has proven to be effective and is relatively economical. However, there are some instances where the addition of aluminum in amounts sufficient to produce a fine grained steel results in an aluminum content in the steel which is detrimental.

Elements such as vanadium, columbium, titanium and zirconium, either singly or in combination, have been used in place of aluminum to produce a fine grain size. The most common addition for grain refining other than aluminum is vanadium but, although vanadium is effective in producing fine grain size, it is relatively expensive. The use of my novel alloy produces a fine grained steel which does not have the drawbacks resulting from the use of aluminum and is considerably less expensive than vanadium.

I have discovered a novel alloy which is effective in producing high quality, fine grained steel in either the cast or wrought condition. My novel alloy may be added without an aluminum addition; or, in some instances, it may be used along with a very small addition of aluminum.

An alloy, according to my invention, which will create fine grained steel without any aluminum addition contains 30-60% silicon, up to 15% manganese, up to 15% calcium plus barium, and 20-40% of at least four of the elements from the group consisting of cerium, lanthanum, columbium, tantalum, vanadium, zirconium, aluminum and boron, with the balance essentially iron and incidental impurities.

While the composition ranges specified above provide alloys which will produce the desired fine grain size, a preferred composition range is as follows:

	Percent
Silicon -----	30-45
Manganese -----	5-10
Calcium -----	1-6
Barium -----	1-6
Ceriumlanthanum -----	5-20
Columbiumtantalum -----	1-6
Vanadium -----	1-6
Zirconium -----	1-6
Titanium -----	1-6
Aluminum -----	1-3
Boron, maximum -----	.04-.15
Iron and incidental impurities -----	Balance

A typical alloy which has been used to produce a fine grained steel is:

	Percent
Silicon -----	40.90
Manganese -----	5.90
Calcium -----	2.12
Barium -----	1.00
Ceriumlanthanum -----	5.32
Columbiumtantalum -----	5.60
Vanadium -----	5.70
Zirconium -----	4.89
Titanium -----	5.63
Aluminum -----	1.00
Boron -----	.06
Iron and incidental impurities -----	Balance

In order to determine the ability of my novel alloy to control grain size in steel, additions were made to heats of AISI 1040 steel. The compositions of the alloys added for grain size control are set forth below in Table I.

TABLE I

Element	Percent	
	Heat 47	Heat 48
Si -----	33.45	40.90
Mn -----	8.07	5.90
Ca -----	2.80	2.12
Ba -----	1.05	1.00
Ce plus La -----	18.02	5.32
Cb plus Ta -----	2.31	5.60
V -----	2.45	5.70
Zr -----	1.49	4.89
Ti -----	2.52	5.63
Al -----	1.05	1.00
B -----	.06	.06
Fe and Incidental Impurities -----	Balance	Balance

The grain refining alloy was added to the steel as it was tapped, and in each instance four pounds of a calcium-ferrosilicon alloy were added per ton of steel for deoxidization. The calcium-ferrosilicon alloy consisted of 16.0% calcium, 57.8% silicon and the balance essentially iron. In various heats small additions of aluminum were also made and the amounts are set forth in Table II. In each instance the aluminum was added as an alloy consisting of 19.0% aluminum, 38.9% silicon and the balance essentially iron.

After the additions were made each ladle was teemed into a 4" x 4" x 24" mold, and the ingots were forged to 1 1/4" square bars. A section of each bar was heat treated and the ASTM grain size determined by the McQuaid-Ehn tests. An ASTM rating of 1 to 5 is considered coarse and an ASTM rating of 5 to 8 is considered fine although grain sizes finer than 8 occur. The terms fine grained steel and fine grain size when used herein refer to a steel having an ASTM rating of 5 to 8 and finer. The grain size results of the individual tests and the ladle additions are set forth in Table II.

TABLE II

Heat No.	Ladle Addition	McQuaid-Ehn Grain Size, 1,700° F.
50C.....	.015% Al plus 1 lb./T.Ht. 47.	7, 8 and finer.
50D.....	.015% Al plus 1 lb./T.Ht. 43.	7, 8 and finer.
51C.....	.010% Al plus 1 lb./T.Ht. 47.	7, 8 few 6's.
54D.....	.010% Al plus 1 lb./T.Ht. 48.	7, 8 few 6's.
58C.....	.005% Al plus 1 lb./T. Ht. 47.	Duplex 6-8 plus 3-4 (5%).
58D.....	.005% Al plus 1 lb./T.Ht. 48.	6-8 with 5's.
57C.....	1 lb./T. Ht. 48.	6-8 with 5's.
49A.....	.015% Al.....	1-4 with 5's.

Heat 49A in Table II was made for comparison and shows that a coarse grain steel is obtained at an aluminum addition of .015% or below when my novel alloy is not added. When using my novel alloy, a fine grained steel was obtained with an addition having a composition within the preferred range of my alloy with aluminum additions of .015% and .010% as shown respectively by Heats 50C and 50D and 54C and 54D. At aluminum additions of only .005% the steel was substantially fine grained with an addition of only one pound per ton of steel of an alloy having the composition of Heat 58C and completely fine grained with the addition of one pound per ton of steel of an alloy having the composition of Heat 58D. As is shown by Heat 57C in Table II, a fine grained steel is obtained without the addition of any aluminum with the addition of one pound of an alloy having the composition of my typical alloy per ton of steel. The amount of each grain refining element added by the addition of one pound of the Heat 48 alloy per ton of steel is less than .003%.

My novel alloy may be made by smelting a rare earth concentrate in a three phase submerged arc furnace using carbon electrodes. Silicon is added to the furnace charge as North Carolina quartzite as such is high in silicon content, but other silicon containing materials may also be used. Although any form of rare earth concentrate such as rare earth oxide or bastnasite concentrate may be used, it is preferable to use the least expensive form of rare earth concentrate that is readily available. The various addition elements are added to the charge as their ores, concentrates or compounds. The following non-limiting example shows the approximate weight of the various raw materials charged to an electric furnace six feet in diameter using 10 inch carbon electrodes to make approximately 330 pounds of my novel alloy in a charge to tap time of about 2 hours. The materials charged and the elements added thereby are as follows.

Material:	Pounds
North Carolina quartzite (Si)	438
Technical vanadium pentoxide (V)	33
Rare earth oxide concentrate (Ce/La)	26
Pyrochlore concentrate (Cb)	28
Zircon sands (Zr)	43
Ilmenite concentrate (Ti)	60

Limestone (Ca)	55
Barium carbonate (Ba)	26
Manganese ore (Mn)	40
Razorite (B)	3
Aluminum ingot (Al)	17
Coal (metallurgical A)	377
Wood chips	142

The novel complex alloy produced from the above charge has a composition within my preferred range.

As stated heretofore, aluminum, vanadium, columbium, titanium and zirconium are known grain refining elements and have been used in various amounts for the production of fine grained steel. However, the total addition of my novel alloy necessary to produce a fine grained steel is less than the amount of any of the single known grain refining elements which must be added to produce fine grain size. It is obvious, therefore, that the combination of these elements in a single alloy exhibits a synergistic effect on the control of grain size. Thus, the addition of one pound of alloy Heat 48 per ton of steel produced a fine grain size, even though the addition of any single known grain refining element was less than 30 p.p.m.

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

I claim:

1. An alloy for the control of grain size in cast or wrought steel consisting essentially of silicon from 30-60%, manganese up to 15%, calcium plus barium up to 15%, a total of 20 to 40% of at least four of the elements from the group consisting of cerium, lanthanum, columbium, tantalum, vanadium, zirconium, titanium, aluminum and boron, and the balance iron and incidental impurities.

2. An alloy according to claim 1 consisting essentially of silicon from 30-45%, manganese from 5-10%, calcium from 1-6%, barium from 1-6%, cerium plus lanthanum from 5-20%, columbium plus tantalum from 1-6%, vanadium from 1-6%, zirconium from 1-6%, titanium from 1-6% aluminum from 1-3%, boron from 0.04-0.15%, and the balance iron and incidental impurities.

3. An alloy according to claim 2 consisting of about 40.90% silicon, about 5.90% manganese, about 2.12% calcium, about 1.00% barium, about 5.32% cerium plus lanthanum, about 5.60% columbium plus tantalum, about 5.70% vanadium, about 4.89% zirconium, about 5.63% titanium, about 1.00% aluminum, about 0.06% boron, and the balance iron and incidental impurities.

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CHARLES N. LOVELL, *Primary Examiner.*

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,383,202

May 14, 1968

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It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

Column 3, line 26, before "Heat" insert -- Ht. 47 as shown in --; line 29, before "Heat", first occurrence, insert -- Ht. 48 as shown in --.

Signed and sealed this 7th day of October 1969.

(SEAL)

Attest:

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Attesting Officer

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Commissioner of Patents