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[54] **METHOD FOR THE PRODUCTION OF HIGH STRENGTH NOTCH TOUGH STEEL**

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Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 253,897, May 16, 1972, abandoned.

[30] **Foreign Application Priority Data**

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[51] **Int. Cl.**[2] **C21D 1/02**

[58] **Field of Search** ..... 148/12 F, 36; 72/364, 365; 29/527.7; 75/124

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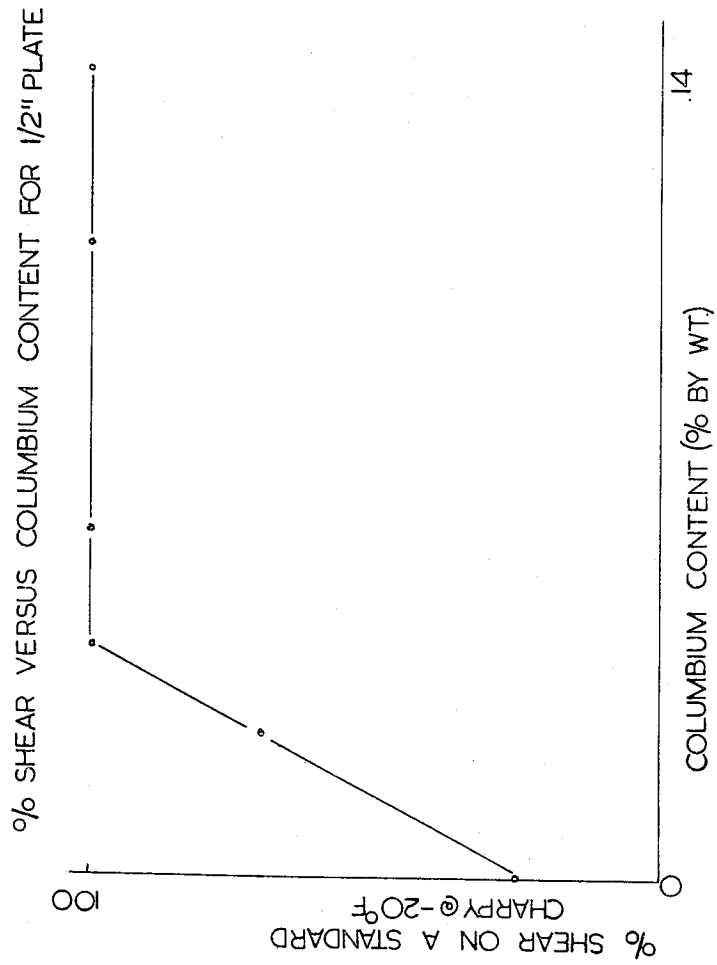
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[57] **ABSTRACT**

A fully killed steel having a high yield strength and high notch toughness below 0°F. and containing by weight up to 0.20 percent carbon, at least 0.015 percent columbium and at least 0.75 percent manganese, said steel having a substantially uniform, fine grain, essentially ferritic grain structure with an average grain size of finer than 9.0 as determined by ASTM Test No. E 112, said steel preferably having a longitudinal Charpy Impact of at least 60, as well as a method of producing said steel which essentially involves continuously rolling the steel in the temperature interval 1700° to 1900°F.

6 Claims, 1 Drawing Figure


% SHEAR VERSUS COLUMBIUM CONTENT FOR 1/2" PLATE
% SHEAR ON A STANDARD CHARPY @ -20°F
100
0
.14
COLUMBIUM CONTENT (% BY WT.)

METHOD FOR THE PRODUCTION OF HIGH STRENGTH NOTCH TOUGH STEEL

CROSS REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of abandoned application Ser. No. 253,897, filed May 16th, 1972.

The present invention relates to steel. In particular the present invention relates to steel of improved yield strength and notched toughness at low temperatures, particularly below 0°F. and a method of making said steel.

There is an ever present and increasing requirement for steels of improved strength and notch toughness and with the development of the arctic regions, such as arctic gas and oil field reserves, steels with high notch toughness at low temperatures and yield strength have become increasingly desirable. For example in the production of pipelines.

Heretofore it has been conventional in producing such steels by subjecting mild steels modified with a suitable grain refiner such as columbium and/or vanadium at levels totalling approximately 0.05 percent by weight of the steel to a controlled rolling process. Conventionally controlled rolling processes involve heating the ingot or slab to a temperature in excess of 2250°F. and rolling the ingot or slab with a rolling schedule involving delays in the pass sequence such that substantial reduction in thickness occurs below a temperature in the range 1700° to 1750°F. finishing with a temperature of approximately 1450°C. Typically these delays are introduced in the temperature range 1700°F. to 1900°F. and are necessary to assure sufficient reduction at the lower temperature to produce a fine grain structure. The degree of reduction required below 1550°F. introduces difficulties in producing flat plate. In addition the properties achieved in the steel are less consistent than desirable due to the necessity of precise temperature and process control. For this reason it is often necessary to normalize the plates produced in this manner especially when the thickness of the rolled plates is ½ inch or greater. This normalizing process is of considerable additional expense both cost-wise and time-wise in the process. Still further, low temperature, notch toughness and strength of low alloy, mild steel plate produced by conventional controlled rolling with or without normalizing is relatively low and substantial improvement is desirable.

The present invention provides an improved steel of substantially higher notch toughness at low temperatures, i.e. below 0°F. which make it highly suitable for structures which are subject to low temperatures, e.g. in the arctic such as pipelines and power transmission towers, which steels may be produced by a simple and more efficient rolling process than the conventional controlled rolling process.

It has now been found that an improved steel plate can be made by rolling an ingot or slab of steel having a low carbon and high columbium content according to a conventional rolling technique provided that the steel is rolled continuously in the temperature interval 1700°–1900°F. and preferably 1700°–1950°F.

According to the present invention therefore, there is provided a method of making a high strength steel of improved notch toughness at temperatures below 0°F. which method comprises subjecting an ingot or slab of

said steel of chemical composition set forth hereinafter and at a temperature of at least 2100°F., preferably at least 2250°F. to a reduced rolling, said rolling being continuously effected through the temperature range 1900°F. to 1700°F.

The present invention also provides a fully killed steel having a high yield strength a high notch toughness below 0°F. and containing up to 0.20 percent by weight carbon at least 0.75 percent manganese and at least 0.015 percent by weight columbium, said steel having an essentially uniformly fine grain essentially ferritic grain structure with an average grain size of finer than 9.0 as determined by ASTM Test No. E 112.

The present invention provides, according to a particular embodiment of the present invention, a fully-killed steel having a high yield strength and high notch toughness below 0°F. and consisting essentially of by weight 0.02 to 0.12 percent carbon, 0.09 to 0.18 percent columbium, 1.0 to 2.0 percent manganese, 0 to 5 percent molybdenum, not more than 0.025 percent phosphorus, not more than 0.035 percent sulphur, 0 to 0.6 percent copper, 0 to 1.0 percent nickel, 0 to 1.0 percent chromium, 0 to 0.2 percent zirconium, 0 to 0.06 percent rare earth metal, the balance being iron and incidental impurities said steel having a substantially uniform fine grain substantially ferritic grain structure with an average grain size finer than 9 as determined by ASTM Test No. E 112.

The carbon is present in the steel in an amount up to about 0.20 percent and preferably from about 0.02 percent by weight to 0.12 percent by weight. In amounts above 0.12 percent by weight the steel tends to lose its high notch toughness due to the formation of substantial amounts of pearlite grains in the structure of the steel and further it is found that the impact strength of the steel is also substantially reduced as is its weldability which is highly desirable in the steel plate. It is also found that the low carbon content raises the A_3 transformation temperature and thus reduces the crystallization of austenite subsequent to the final rolling of the steel. The lower the carbon content of the steel, the better the notch toughness. The manganese content of the steel however is usually provided by means of a ferro manganese addition containing carbon as an impurity and there is a practical lower limit for the carbon present in the steel. Preferably the carbon is present in an amount from 0.04 to 0.10 percent and more preferably 0.06 to 0.07 percent by weight. It has been found that the fine grain structure in the steel which is predicated primarily on the columbium content is still achieved with steels having a carbon content up to about .20 percent despite the formation of pearlite grains in the structure and the steel still has acceptably high impact strength and percentage shear, particularly when the carbon content is below 0.16 percent. However, to maintain weldability with such high carbon content in the steel, the columbium content must be reduced and thus a high carbon content in the steel which lowers impact strength and percentage shear in the steel and a low columbium content which also lowers the impact strength and percentage shear in the steel is not preferred.

The columbium is preferably present in the steel in an amount from 0.09 to 0.18 percent and more preferably in the range 0.12 to 0.15 percent by weight of the steel. It is believed the net result of the low carbon content and the higher than conventional columbium is to

provide a volume fraction of columbium carbide and columbium carbo-nitride precipitates in the steel sufficient to retard austenite grain growth at high temperatures. It is believed that the combination of low carbon content and high columbium content in the steel affords a greater potential for the precipitation of columbium carbo-nitride and columbium carbide in the temperature range 1700°F. to 1900°F. because the solubility limit of columbium in austenitic steel increases with decrease of carbon content, i.e. a greater volume fraction of the precipitate of columbium carbide and columbium carbo-nitride is present at a given temperature when the carbon content of the steel is decreased and the columbium content increased. It is thus found that by continuously reducing the steel in the temperature interval 1700°F. to 1900°F. by a rolling schedule such as to produce 50 percent reduction in the thickness of the steel below about 1800°F. a very fine essentially uniform grain size is achieved and maintained. With a columbium content of less than 0.09 percent by weight the degree of grain refinement which is capable of being achieved in the austenite is reduced. It is possible however to achieve the fine grain structure steel by the rolling technique of the present invention with substantially less columbium than 0.09 percent with consequent high impact strength and high percentage shear that is required. In particular, it has been found that the columbium content may be as low as 0.015 percent by weight and still achieve the fine grain structure with high percentage shear and high impact strength. Suitably, the columbium content is at least 0.04 percent and more suitably at least 0.06 percent by weight. It is found that the impact strength and percentage shear increase with percentage columbium and above about 0.06 percent it is believed the colubmium acts inter alia to increase ultimate tensile strength.

The manganese is present in the steel to provide strength including both yield and ultimate tensile strength and in particular the manganese is believed to provide strength in the ferrite grains in the steel. The strength of the steel is determined to some extent by the thickness of the steel plate and the amount of manganese present in the steel. In general the thicker the steel the lower its yield strength and ultimate tensile strength for a particular chemical composition. By raising the manganese content of the steel, the strength of the steel is thus increased. In general it has been found for thin steel plate of less than 0.42 inches to obtain a high strength steel with a yield strength in excess of about 60,000 psi the manganese content should preferably be in excess of about 1 percent and for thicker steel should be at least 1.0 to 2.0 percent by weight. However, a manganese content of the steel may be as low as about 0.75 percent and still achieve the advantages of the present invention. A manganese content below 0.75 percent yields a steel which tends to have a low percentage shear. The maximum manganese contents is determined to a great extent by the carbon equivalent required in the steel for weldability. Preferably the manganese content of the steel for thin steel plate is in the range 1.30 to 1.70 percent by weight and more preferably 1.30 to 1.45 percent by weight and for thicker steel in excess of 0.42 inches the manganese content is in the range 1.60 to 2.0 percent by weight. Where it is desired to increase the ultimate tensile strength molybdenum will also be present in the steel, suitably in an amount not in excess of about 0.5 percent by weight. The molybdenum is found to increase the ultimate tensile strength but to slightly decrease the yield strength of the as-rolled steel. The presence of molybdenum is particularly useful in thicker plates to increase the ultimate tensile strength.

Sulfur is present as an impurity in steels and lowers the transverse impact strength of the steel and, in particular, combines with the manganese present in the steel to form manganese sulphide stringers which extend longitudinally in the steel causing directionability of impact properties. The transverse impact strength is increased by adding rare earth metals which are capable of reacting with sulfur in the molten steel. Rare earth metals also react with oxygen in the steel, and the steel is killed suitably by the addition of killing agents such as aluminum or by the addition of larger amounts of the rare earth than are necessary to react with the sulfur. The rare earth thus can be used both as a killing agent and for combination with the sulfur. Desirably the rare earth metals are present in amounts up to 0.06 percent by weight of the steel. Zirconium at levels up to 0.20 percent and preferably up to 0.10 percent may be used in place of rare earth metals as a sulphide modifier and/or a killing agent. Aluminum is the most useful killing agent and is present in an amount sufficient to kill the steel. When too little aluminum is added, the steel is not killed and when too much aluminum is present surface problems may occur in the killed steel. The aluminum is suitably present in the steel as killing agent in an amount from 0.02 to 0.10 percent by weight and preferably from 0.03 to 0.06 percent by weight. The sulfur content of the steel is maintained below 0.035 percent by weight and desirably below 0.020 percent by weight.

Phosphorus is also an impurity in the steel and tends to render the steel brittle and the content of the phosphorus in the steel is maintained below 0.025 percent by weight and desirably below about 0.015 percent by weight.

The steel may also contain conventional strengthening ingredients such as copper, nickel and chromium or mixtures thereof in levels up to 1.0 percent each. Preferably the copper is present in an amount from 0.2 percent by weight to 0.6 percent by weight and preferably up to 0.4 percent by weight. A further such agent which may be mentioned is vanadium which may be present in an amount up to about 0.12 percent by weight.

The rolling of the steel for ingot or slab to plate may be effected on roughing mills and finishing mills with the transfer between the mills being effected at temperatures outside the interval 1900°F. to 1700°F. Alternatively the rolling can be effected on a single mill. It is a critical feature of the present invention that the steel be continuously rolled in the temperature interval from about 1900°F. to about 1700°F. and preferably about 1950°F. to about 1700°F. It has been found still further preferable to roll continuously until the steel temperature has reached 1650°F. It is further stated in the parent application Ser. No. 253,897 that in the continuous rolling that there should be no undue delay and that the transfer between the roughing mills and the finishing mills should be within the aforesaid temperature interval. It should be explained however that provided the transfer between the mills is rapid, e.g. of the order of less than about 20 seconds, the transfer may take place at temperatures down to about 1850° F. without de-

tracting significantly from the fine grain structure and impact and percentage shear of the steel produced.

In the rolling of the steel the initial temperature of the ingot may be at least 2100°F. and preferably at least 2250°F. in order to ensure that the columbium is maintained in solution in the austenitic steel, and allows the columbium to precipitate out as columbium carbide and/or columbium carbonitride particles at a temperature in the range 1700° to 1900°F. when continuous rolling of the steel causes the columbium carbide and/or columbium carbonitride to precipitate and thus refine austenitic grain structure. Thus it is a critical feature of the present invention that the slab be continuously rolled in the temperature range 1900°F. to 1700°F. and preferably 1950° to 1700°F. with no undue delay between passes. Desirably there will be at least 50 percent reduction of the thickness of the steel plate at a temperature below 1800°F. It is believed that a conventional steel rolled according to the known controlled rolling process cannot produce and maintain a fine austenitic grain size above 1700°F. The rolling of the conventional steel from 1700°F. down to about 1450°F. serves to develop a fine grain structure in the rolled steel plate. In contrast thereto with the rolling reduction process of the present invention in combination with the low carbon content of the steel the formation of pearlite during cooling of the steel is substantially reduced and the high columbium content of the steel allows for fine grain particles of columbium carbide and/or columbium carbonitride (the nitrogen being present as a natural impurity in the steel). By continuously rolling the steel in the range 1700° to 1900°F. and preferably 1700° to 1950°F. this ensures that the columbium carbide and/or columbium carbonitride particles precipitate out and provide a substantially uniformly fine essentially ferritic grain steel having improved notch toughness at low temperatures below 0°F. and also below −50°F.

The finishing temperature of the steel in the process of the present invention is usually substantially higher, e.g. in the range 1500° to 1600°F. thus making the production of flat steel plate simpler than in the conventional controlled rolling process. However it is within the scope of the present invention to continue the rolling to lower temperatures as in the controlled rolling process to improve the toughness and strength properties of the steel plate even further.

It has been found that the steel produced by the present invention has a very fine grain structure which is substantially uniform with an average grain size of finer than 9 and preferably at least 9.5 and more, at least 10 as determined by ASTM Test No. E112 and it is found that this uniform fine grain size is present in thin rolled plate steel of thickness of about ⅛ inch and in thicker rolled plate steel in the range ½ inch to about 1⅛ inches. It is believed that it is this uniform fine grain ferritic structure in the steel which contributes particularly to its high notch strength particularly at low temperatures in the range 0°F. to −50°F. Thus it will be seen from the following Examples that at temperatures below 0°F. that the steels have longitudinal Charpy Impact Values of at least 60 ft. lbs., preferably at least 80 ft. lbs., more preferably at least 190 ft. lbs. and even more preferably at least 200 ft. lbs. with a percentage shear of at least 90 percent, and transverse Charpy Impact Values of at least 60 ft. lbs. at 0°F., at least 50 ft. lbs. at −20°F. and at least 40 at −50°F.

The present invention will be further illustrated by way of the following Examples.

EXAMPLE 1

Three plates were prepared from an electric arc furnace heat of the following composition:

Element	Amount
Carbon	0.07%
Manganese	1.25%
Sulphur	0.021%
Phosphorus	0.010%
Silicon	0.02%
Columbium	0.14%
Aluminum	0.060%
Iron and Impurities	Balance

Three slabs 54 inches × 7 inches × 86 inches were rolled in a 2 HI roughing mill and then in a 4 HI finishing mill to a final (as rolled) plate size 102 inches × 5/8 inch × 447 inches according to the schedule set forth in the following Table 1.

Of these plates, plate 544 which was continuously rolled in the temperature range 1500°–2040°F. has the best combination of properties, maintaining 83 ft. lbs. impact energy with 95 percent shear in the fracture surface down to −50°F.

Plate 545 was essentially continuously rolled from 2200°F. down to 1760°F. Although good impact properties were measured at 0°F., the percent shear area is lower at all temperatures as compared to plate 544.

Plate 541 was not rolled in the temperature range 1857°–1942°F. and displayed impact properties inferior to those of plates 544 and 545.

Table 1

Plate No.	2 HI Mill Entry Temp.	Exit Temp.	4 HI Mill Entry Temp.	Exit Temp.	Tensile Results Yield (psi)	U.T.S. (psi)	Elongation(% in 2")
541	2225	1942	1857	1730	62,900	74,500	38
544	2215	2103	2040	1500	66,600	74,000	37
545	2200	1850	1845	1760	62,900	72,500	40

	Charpy Impact Values								
	Longitudinal 0°F.		−20°F.		−50°F		Transverse 0°F.	−20°F.	−50°F.
Plate No.	ft. lb.	% shear	ft. lb.	% shear	ft. lb.	% shear	ft. lb.	ft. lb.	ft. lb.
541	51	40	40	15	21	5	19	16	8
544	83	95	84	95	83	95	30	27	23
545	94	90	89	90	75	80	30	25	19

EXAMPLE 2

Five plates of approximately ½ inch thickness were prepared from a basic oxygen steel plant heat of the following composition:

Element	Amount
Carbon	0.10%
Manganese	1.23%
Silicon	0.04%
Sulphur	0.020%
Phosphorus	0.007%
Columbium	0.11%
Aluminum	0.050%
Iron and Impurities	Balance

Five slabs of the aforesaid composition were rolled on a 2HI roughing mill and then on a 4 HI finishing mill. The rolling temperature schedule used and the properties obtained are summarized in the following Table 2.

It will be seen from Table 2 that the plates which were all continuously rolled in the temperature interval 1900°F to 1700°F had Charpy Impact values in excess of 80 ft. lbs. and a percent shear of at least 90 percent.

The steel slabs were first rolled in a 2 HI roughing mill and then on a 4 HI finishing mill to a final (as rolled) plate size of 139 inches × 0.42 inches × 770 inches according to the schedule set forth in Table 3.

The tensile strength and the Charpy Impact Values of the steel plate obtained are shown in Table 3. The substantially improved Charpy Impact Values of the steel plates are those of Example 3 are believed to be due to the presence of the rare earth metals (e.g. cerium) which reduces the sulphur content of the steel and reduces the effect of the sulphur remaining in the steel.

The steel had a substantially uniform ferritic grain structure of very fine grain size of 12 as determined by ASTM E 112.

Table 2

Plate No.	2HI Entry Temp.	2HI Exit Temp.	4HI Entry Temp.	4HI Exit Temp.	Gauge	Yield ft.(psi)	U.T.S. (psi)	Elong. (% in 2")	Longitudinal Impact ft. lb.	Results at −20°F. % shear
39714	2250	2040	1976	1526	.455"	64,200	75,190	24	84	90
39715	2250	2045	1912	1509	.465"	64,650	74,770	25	88	90
39716	2250	2023	1916	1518	.500"	65,100	73,460	24	91	95
39720	2250	2000	1914	1506	.455"	65,790	76,130	23	93	90
39721	2250	2040	1914	1505	.460"	65,860	75,550	25	96	90

Table 3

Plate No.	2 HI MILL Entry Temp.	2 HI MILL Exit Temp	4 HI MILL Entry Temp.	4 HI MILL Exit Gauge	Temp	TENSILE RESULTS Yield (psi) Long	Yield (psi) Trans	UTS (psi) Long	UTS (psi) Trans	Element iv 2"% Long	Element iv 2"% Trans
4858	2180	1925°	1894	0.429"	1530	67,600	67,900	78,200	79,000	41	42
4859	2180	1945	1889	0.424"	1529	70,300	71,400	80,300	80,500	39	39
4860	2180	1924	1892	0.428"	1525	66,100	68,000	76,600	71,200	38	40
4861	2180	1967	1910	0.429"	1550	69,500	69,200	79,100	79,500	39	39
4861	2175	1930	1899	0.429"	1540	69,400	20,900	80,000	80,100	41	41

Plate No.	Charpy Impact Values Longitudinal 0°F. Ft.Lb.	0°F. %Shear	Longitudinal −20°F. Ft.Lb.	−20°F. %Shear	Longitudinal −50°F. Ft.Lb.	−50°F. %Shear	Transverse 0°F. Ft.Lb.	0°F. %Shear	Transverse −20°F. Ft.Lb.	−20°F. %Shear	Transverse −50°F. Ft.Lb.	−50°F. %Shear
4858	203	95	203	90	189	90	69	40	52	20	45	20
4859	209	100	212	95	211	95	111	80	100	75	74	25
4860	195	95	196	95	201	90	78	85	60	85	71	55
4861	206	95	201	95	188	90	79	65	76	60	57	25
4862	220	100	223	95	226	95	94	50	87	45	75	25

EXAMPLE 3

Five steel plates were prepared from a basic oxygen steel plant heat with a slab size 74 inches × 7 inches × 102 inches of a steel of the following composition:

Element	Amount
Carbon	0.07%
Manganese	1.35%
Silicon	0.08%
Sulphur	0.010%
Phosphorus	0.009%
Columbium	0.13%
Aluminum	0.06%
Cerium	0.021%
Iron and Impurities	Balance

EXAMPLE 4

Table 4 presents the temperature schedule of three plates of the same composition noted in Example 2.

Plate 31068 was rolled in the manner of a conventionally controll rolled steel with the resultant high yield and ultimate tensile strength noted.

Plate 39716 was rolled in a manner consistent with continuous reduction in the temperature range 1700°–1900°F. with a finishing temperature considerably higher than plate 31068.

Plate 39087 represents a plate rolled with no controlling restrictions placed on the 4 HI mill.

Table 4 shows the definite improvement in the impact properties achieved in plate 39716.

It was observed that plate 39716 had the best microstructure, being completely free of the undesirable bands of coarse grain observed in the other plates.

Table 4

Pl. No.	2 HI Entry Temp.	2 HI Exit Temp.	4 HI Entry Temp.	4 HI Exit Temp.	Gauge (in.)	Yield (psi)	U.T.S. (psi)	Elong.	Long. Impact at −20°F. ft. lb.	Long. Impact at −20°F. % shear
39087	2250	2035	1899	1800	.560	64400	76020	34% (2")	27	95
39716	2250	2023	1916	1518	.500	65100	73460	24% (8")	91	95
31068	2250	2020	1737	1387	.475	77580	87700	16% (8")	72	95

EXAMPLE 5

18 inches × 7½ inches × 40 lb/ft. wide flange structurals were produced from a basic oxygen steel plant heat of the following compositions:

Element	Amount
Carbon	0.07%
Manganese	1.39%
Silicon	<0.10%
Sulphur	0.018%
Phosphorus	0.008%
Columbium	0.13%
Aluminum	0.050%
Cerium	Nil
Iron and Impurities	Balance

A 22 inches × 15 inches bloom was rolled into 22½ inches × 11 inches × 4 inches blanks which were reheated to 2100°F., broken down in a breakdown mill and then transferred to a parallel flange mill where it was rolled continuously through the temperature range 1600°–1950°F.

Table 5

	Flange	Web
Yield strength (psi)	72,620	75,540
Ultimate Tensile Strength (psi)	80,360	84,860
Elongation (% in 8")	20	23
Longitudinal Impact Results (⅔ Size Charpy Specimens at 0°F)		
Ft. Lb.	231	195
% Shear	100	100

While the present invention has been described with reference to plate production it also has application to the production of sheet products in general and rolled shapes such as structural products, e.g. I-beams, wide flange beams and merchant shapes.

The temperatures referred to herein throughout the disclosure and claims refer to the mean temperature of the steel being rolled and not the surface temperature of the steel which is typically 50°F to 75°F lower than the mean temperature. In the Examples herein, the mean temperature of the steel was calculated by computer based on the mean reheat soaking temperature which can be fairly accurately determined. The computer makes adjustments to the calculated mean temperature of the steel at any particular place in the rolling procedure based upon hardness mill operating parameter such as rolling speeds, measured wall forces (e.g. hardness of the steel forming the plate) water sprays, etc. Such computer control of the rolling procedure and the calculation of the mean temperature of the steel from the rolling history and properties of the steel plate are known techniques inter alia to Canadian General Electric Company and do not form part of the present invention.

Operating the process of the present invention using pyrometers which read the surface temperature of the steel only it is possible to calculate the mean temperature of the steel based on the prior history of the rolling procedure of the steel usually to within an error of ±20°F with the pyrometers properly set up and calibrated. Thus, in rolling mills operating with pyrometers, i.e. without computer control may operate in accordance with the present invention by making appropriate adjustments to the critical temperature range as measured by the pyrometers such as for example continuously rolling in the temperature range about 1850°F to about 1650°F based on the assumption that there is 50°F temperature difference between the surface temperature of the steel and the mean temperature of the steel.

EXAMPLE 6

Four plates of the thickness set forth in the following Table 6A were prepared from four basic oxygen steel plant heats of the composition set forth in the following Table 6.

Table 6

	Composition % by weight						
Heat No.	C	Mn	S	P	Cb	Al	Fe and Impurities
D 7085	0.14	1.38	0.008	0.009	0.060	0.065	balance
F 1403	0.15	1.42	0.010	0.007	0.060	0.060	balance
F 1057	0.14	1.34	0.008	0.009	0.060	0.059	balance
F 1237	0.15	1.58	0.009	0.013	0.062	0.042	balance

The steel in each of the heats was rare earth treated so as to contain sufficient rare earth metal values to achieve 70 to 100 percent sulphide modification.

A slab of each of the aforesaid heats was rolled on a 2HI roughing mill and then a 4HI finishing mill. The slab entering the 2HI roughing mill was 8.9 inches thick, entered the mill at a temperature of 2234°F and was rolled to a thickness of 3.4 inches exiting from the mill at a temperature of 1934°F. The 3.4 inch thick plate was then immediately passed to the 4HI finishing mill entering at a temperature of 1876°F and was rolled to the final thickness shown in Table 6A exiting at a temperature of 1645°F.

The results obtained are shown in Table 6A.

Table 6A

HEAT NO.	PLATE NO.	YIELD (PSI)	U.T.S. (PSI)	% ELONG (8")	CHARPY IMPACT RESULTS LONGITUDINAL FT. LBS.	LONGITUDINAL % SHEAR	TRANSVERSE FT. LBS.	TRANSVERSE % SHEAR	TEST TEMP. (°F)	PLATE THICK (IN)
F1237	19139	68570	84410	21	115 176 153	100 100 100	62 51 54	50 40 40	−20	⅝
F1403	19998	68340	86060	25	116 123	65 65 65	54 74 68	95 95 95	−20	⅝
D7085	14497	62800	79400	26	113 106 125	100 100 100	78 93 103	100 100 100	0	¾
F1057	14498	61800	77570	25	195 145 137	100 100 100	100 100 100	100 100 100	0	¾

EXAMPLE 7

In a similar manner as in Example 6 plates 0.500 to 0.75 inches thick were rolled from a basic oxygen steel plant heat slabs of the following composition:

Element	Amount (% by weight)
Carbon	0.10
Manganese	1.20
Sulphur	0.009
Phosphorus	0.007
Silicon	0.080
Aluminium	0.060
Vanadium	0.040
Columbium	0.040
Iron & Impurities	Balance

Each heat was rare earth treated in a similar manner to Example 6. Each heat was continuously rolled for reduction from 1910°F to a finishing temperature of approximately 1550°F with a greater than 50 percent reduction below 1800°F.

The plates so obtained had the following properties.

Physical Properties (Heat Average)
Yield Strength — 66,500 psi
Ultimate Tensile Strength — 78,400 psi
Elongation — 32% in 2 inches
Impact Properties (For full size Charpy specimens)

		0°F	−20°F	−40°F	−80°F	−100°F	−140°F
Longitudinal	Ft.lbs.	219	207	177	170	150	91
	% Shear	100	100	95	80	65	23
Transverse	Ft.lbs.	*N.T.	118	116	94	100	*N.T.
	% Shear	*N.T.	73	72	42	49	*N.T.

Microstructure:
Grain size (.75" Plate) Surface ASTM No. 10.5
Core ASTM No. 10

*N.T. — not tested

EXAMPLE 8

In a similar manner to Example 7 plates 0.500 inches thick were rolled by precisely the same schedule as in Example 7 from slabs of a heat of the following composition:

Element	Amount (% by weight)
Carbon	0.100
Manganese	0.94
Sulphur	0.028
Phosphorous	0.009
Silicon	0.240
Chromium	0.540
Nickel	0.290
Copper	0.340
Columbium	0.025
Aluminium	0.040
Iron & Impurities	Balance

The heat was not rare earth treated. The plate obtained had the following properties.

Physical Properties
Yield Strength — 64,400
Ultimate Tensile Strength — 80,150
Elongation — 20% in 8"
Impact Properties (For full size Charpy specimens)

		0°F	−20°F	−50°F	−60°F
Longitudinal	Ft.lbs.	82	62	29	30
	% Shear	95	70	30	5

Microstructure
Grain Size (½" Plate) Surface ASTM No. 12.5
Core ASTM No. 12.0

On rolling a slab of similar composition except for the absence of columbium according to the same schedule the plate obtained had the following properties.

Impacts - 31 ft. lb. and 25% shear −20°F
Grain Size (½" Plate) - Surface -ASTM 8.0 Core - ASTM 8.0.

It will be seen that the presence of columbium is essential for the production of the steel of the present invention of fine grain size.

The Figure represents a graph for ½ inch plate of columbium content of the steel against percentage shear in a standard Charpy sample broken at −20°F, the results being taken from the preceeding Examples. It will be seen that the rolling process of the present invention is effective to some degree on steel at columbium levels from 0 to 0.14 percent. For optimum results the steel of ½ inch thickness should contain at least 0.04 percent columbium. This optimum columbium level varies with plate thickness being higher for thinner plates and vice versa.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. A method of making a high strength steel of improved notch toughness below 0°F which comprises subjecting a fully killed steel consisting essentially of the following composition by weight:

carbon	0.02	to	0.20%
manganese	0.75	to	2.0%
columbium	0.015	to	0.18%
aluminum	0.02	to	0.10%
nickel	0	to	.0 %
chromium	0	to	1.0 %
copper	0	to	1.0 %
molybdenum	0	to	0.5 %
vanadium	0	to	0.12%
zirconium	0	to	.20%
rare earth metals	0	to	.06%
balance	iron and impurities		

to heating to at least 2100°F, and subsequently rolling said steel to continuously reduce the steel, without any substantial time delays, throughout the temperature range of from 1900° to 1700°F with at least a 50 percent reduction in section at below 1800°F, said steel having a uniform fine grain of an essentially ferritic grain structure with an average grain size of finer than 9.0 as determined by ASTM No. E 112.

2. A method as claimed in claim **1** in which the rolling is continuous throughout the temperature range about 1950°F to about 1700°F of the steel.

3. A method as claimed in claim **1** in which the reducing rolling is effected in both a roughing mill and

subsequently in a finishing mill, the transfer of the steel from the roughing mill to the finishing mill being above or below the temperature range 1900°F to 1700°F of the steel.

4. A method as claimed in claim 1 in which the initial temperature of the ingot is at least about 2250°F.

5. A method as claimed in claim 1 in which the rolling is continuous throughout the temperature range about 1900°F to about 1650°F of the steel.

6. A method as claimed in claim 1 in which the rolling is continuous throughout the temperature range about 1950°F to about 1650°F of the steel.

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