

[54] **HOT-ROLLED HIGH-STRENGTH  
LOW-ALLOY STEEL AND PROCESS FOR  
PRODUCING SAME**

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[58] **Field of Search**..... **148/12, 36; 75/123 J**

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[57] **ABSTRACT**

A ferritic hot-rolled high-strength low-alloy steel having a yield strength in excess of 65 ksi and excellent subzero impact properties which contains 0.03 to 0.15% carbon, 0.5 to 2.0% manganese, 0.1 to 0.40% molybdenum and 0.01 to 0.10% columbium. The steel is strengthened by the combined effects of grain refinement, precipitation hardening and a high dislocation density which are effected by partially hot rolling the steel above the Ar<sub>3</sub> transition temperature and then hot rolling to effect a 10 to 40% thickness reduction at intercritical temperatures between the Ar<sub>3</sub> and Ar<sub>1</sub> transition temperatures. Some embodiments of this steel may be further strengthened by a subcritical temper, or a small amount of cold working.

**16 Claims, No Drawings**

## HOT-ROLLED HIGH-STRENGTH LOW-ALLOY STEEL AND PROCESS FOR PRODUCING SAME

### BACKGROUND OF THE INVENTION

The stringent demands for increased strength and improved notch toughness in hot-rolled, high-strength, low-alloy steels has stimulated considerable research activity in the areas of alloy development and processing controls. Particularly since the discovery of vast natural gas deposits in Alaska, there has been considerable interest in developing steels with even more stringent strength and toughness requirements for Arctic line-pipe applications.

The well established techniques of grain refinement and precipitation strengthening through composition and processing controls has been refined in recent years so that hot-rolled steels with conventional polygonal ferrite microstructures have appeared to reach a limiting combination of strength and toughness. Although low-temperature controlled-rolling of steel plate is now commonly used in the production of high-strength low-alloy type steels, it has been generally recognized that the yield strength of such steels remains essentially unaffected by the finish-rolling temperature whereas impact notch toughness, particularly impact transition temperature, is continually and markedly improved as the finish-rolling temperature is lowered within the single phase austenite region. It has been recognized that finish rolling at too low a temperature, i.e. below the upper critical ( $A_{r3}$ ) austenite-to-ferrite transformation temperature produces some increase in yield strength but adversely affects impact toughness due to the presence of "cold worked" or unrecrystallized ferrite grains. It has also been shown that hot rolling in the inter-critical austenite plus ferrite phase region can produce undesirable recrystallization and grain growth of ferrite that can adversely affect both yield strength and impact notch toughness.

### SUMMARY OF THE INVENTION

This invention is predicated on my development of a new improved hot-rolled, high-strength, low-alloy steel having yield strengths of 65 to 100 ksi in combination with subzero Charpy V-notch (CVN), 50 percent shear fracture appearance transition temperatures (FATT), which are developed by combining the traditional precipitation-strengthening and grain-refining mechanisms with dislocation strengthening.

Accordingly, it is an object of this invention to provide a new and improved hot-rolled, high strength, low-alloy steel having an exceptional combination of high-strength and low-temperature toughness thereby being particularly suitable for subzero Arctic line-pipe applications.

Another object of this invention is to provide a new and improved hot-rolled, high-strength, low-alloy steel having a yield strength of 65 to 100 ksi and exceptional toughness at subzero temperatures characterized by 50 percent shear FATT values at temperatures as low as  $-80^{\circ}\text{F}$ .

A further object of this invention is to provide a low-carbon, low-alloy steel that can be processed by a special hot working technique to provide a combination of high-strength and good toughness in the as hot-worked condition, and which may be tempered or cold worked in some instances to produce further strengthening

without attendant severe reductions in ductility or impact notch toughness.

Another object of this invention is to provide a high-strength, low-alloy steel containing manganese, molybdenum and columbium which can be processed to provide an exceptional combination of strength and toughness through the combined mechanisms of precipitation strengthening, grain refining and dislocation strengthening.

Still another object of this invention is to provide a process for producing a hot-rolled, high-strength, low-alloy steel having an exceptional combination of strength and toughness which is achieved by controlling alloy composition and controlled rolling practices.

Yet another object of this invention is to provide a process for producing a hot-rolled, high-strength, low-alloy steel utilizing a controlled rolling practice that combines the traditional precipitation-strengthening and grain-refining mechanisms with dislocation-strengthening that is introduced by continuing the hot-rolling operation below the upper critical ( $A_{r3}$ ) temperature, and which may further involve tempering to improve yield strength without severe reductions in ductility or toughness.

Still a further object of this invention is to provide a new and improved steel line-pipe having a yield strength of 65-100 ksi and exceptional toughness at subzero temperatures which is therefore ideally suited for Arctic applications.

Another object of this invention is to provide a low-carbon, low-alloy steel plate which can be fabricated into line-pipe using the conventional U and O process which will exhibit an increase in yield strength after such fabrication.

### DESCRIPTION OF THE PREFERRED EMBODIMENTS

As noted above, this invention concerns a new and improved low-carbon, low-alloy steel which is processed by a special hot working technique to provide a combination of high strength and toughness, which results from a combination of precipitation-strengthening, grain-refining and dislocation-strengthening.

The alloy of this invention has the following composition by weight:

|            | Broad         | Preferred     |
|------------|---------------|---------------|
| Carbon     | 0.03 to 0.15% | 0.05 to 0.10% |
| Phosphorus | 0.04 % max.   | 0.04 % max.   |
| Sulfur     | 0.04 % max.   | 0.04 % max.   |
| Manganese  | 0.5 to 2.0 %  | 1.0 to 1.6 %  |
| Molybdenum | 0.1 to 0.40%  | 0.15 to 0.40% |
| Columbium  | 0.01 to 0.10% | 0.02 to 0.05% |
| Vanadium   | 0 to 0.20%    | nil           |

with the balance comprising iron and conventional impurities. In addition, the steel may be deoxidized with aluminum, silicon or both, and if desired, the alloys of this invention may also contain additional elements such as nickel, copper and/or chromium which may be added as strengtheners or to impart corrosion resistance.

As already noted, the alloys of this invention derive their unique combination of strength and toughness from a novel hot-rolling technique which optimizes three factors, (1) the strengthening effect of a fine po-

lygonal ferrite grain size, (2) the precipitation of fine carbides and/or nitrides within the ferrite, and (3) a high dislocation density that is retained and stabilized by the precipitates. To achieve these ends, a steel slab or other form, having the above composition, must be hot rolled. As in most prior art practices the steel to be hot rolled is heated to a temperature sufficient to dissolve all carbides and nitrides into an austenite matrix. For the above composition, this will require heating the steel to a temperature above 2000°F. After a homogeneous austenitic microstructure is achieved, i.e. by heating above 2000°F, hot rolling of the steel is commenced either at the maximum heating temperature above the  $A_{r3}$  austenite-to-ferrite transformation temperature which is sufficient to retain the carbides and nitrides in solution. The crux of this inventive process resides in not completing the hot rolling at these temperatures, but rather completing no more than about 90% of the intended hot reduction above the  $A_{r3}$  transformation temperature. Thereafter, the partially hot rolled steel is allowed to cool to a temperature below the  $A_{r3}$  transformation temperature, but above the  $A_{r1}$  transformation temperature so that a portion but not all of the austenite is transformed to ferrite. Thereafter, the final hot rolling is performed on the partially transformed metal to effect at least 10% thickness reduction but not enough to cause any recrystallization and/or grain growth of the ferrite grains, i.e., usually not more than about a 40% thickness reduction. Ideally, this intercritical final deformation should provide a thickness reduction within the range 20 to 30%. Thickness reductions of less than 10% will not usually uniformly strain the metal throughout its cross-sections and, therefore, the deformed ferrite grains and the strengthening effect caused thereby will not be uniformly distributed throughout. The upper limit of 40% thickness reductions is somewhat arbitrary, with the actual limit being dependent upon strength of the rolling mill and the ability of the steel to withstand deformation without recrystallization. My experience has shown however that a maximum limit of about 40% thickness reductions is reasonable from practical and metallurgical view points.

It should be apparent that the strengthening effect caused by the deformed ferrite will be a function of the amount of such ferrite present in the steel. Therefore, it is preferred that the steel be cooled to a temperature at least about 25°F below the  $A_{r3}$  transition temperature for the intercritical hot rolling, in order to assure a significant proportion of ferrite in the steel. The lower the temperature the steel is cooled below the  $A_{r3}$  transition temperature, the greater the amount of ferrite formed, and therefore, the higher the resulting yield strength will be.

Upon completion of the intercritical hot rolling between the  $A_{r3}$  and  $A_{r1}$  transition temperatures, the steel is allowed to cool to ambient temperatures where the microstructure is characterized by the presence of both equiaxed and "cold-worked" grains of ferrite in a proportion depending upon the extent of austenite-to-ferrite transformation prior to the final deformation at temperatures below the  $A_{r3}$  temperature, plus a small amount of pearlite and/or bainite. The equiaxed grains of course result from the transformation of austenite after the rolling is completed and the steel cooled. The cold-worked ferrite grains are elongated in appearance

and contain a high dislocation density, and carbide and nitride precipitates appear uniformly distributed within both types of ferrite grains. In this as hot-rolled condition, the steel will exhibit yield strengths of at least 65 ksi, and Charpy V-notch 15 mil lateral expansion transition temperatures (LETT) of from -90° to -150°F, and 50% shear fracture appearance transition temperatures (FATT) of from -45° to -90°F.

Although the unique combination of strength and toughness does indeed result primarily from the above described hot rolling technique, it should be understood that the alloy composition limits are equally critical to achieve the desired end product. In fact, when other low-alloy compositions have been rolled in the intercritical austenite-plus-ferrite phase region, undesirable recrystallization and grain growth of the deformed ferrite grains usually results and has an adverse effect on both yield strength and toughness.

In considering the alloy composition, it is first noted that the carbide and nitride forming elements molybdenum and columbium are of course essential ingredients for conventional precipitation strengthening. In addition, the carbide and nitride precipitate particles must be formed during hot rolling and/or during transformation so that the dislocations and subgrain boundaries in the deformed ferrite grain structure can be pinned and stabilized thereby preventing recrystallization and grain growth to retain the strengthening effect of the dislocations. The mere presence of the carbide and nitride formers alone is not enough however to prevent recrystallization and grain growth. That is, in addition to inclusion of molybdenum and columbium for a stabilizing effect, the carbon and manganese contents, and to some extent the amounts of the carbide and nitride formers, must be critically controlled in order to control the austenite-to-ferrite transformation temperature,  $A_{r3}$ , so it is not so low as to make processing difficult at temperatures therebelow for the final hot rolling step, or not so high as to cause the deformed ferrite grains to recrystallize and grow in spite of the carbide and nitride precipitates. To this end, the composition limits must be adjusted to provide an  $A_{r3}$  transition temperature within the range 1350° to 1500°F, and preferably between 1400° and 1475°F. Quantitatively, increasing either the carbon or manganese contents will tend to decrease the  $A_{r3}$  transition temperature. Accordingly, a good balance between these two elements is preferred. That is, if an exceptionally low carbon content is provided, manganese contents towards the high end of the range are preferred, and vice versa. Since the carbon content of the alloy is preferably kept low at about 0.08% or slightly therebelow to insure good weldability and formability and to also insure the dissolution of carbides into the austenite upon initial heating prior to hot rolling, it is then preferred that for these carbon levels, the manganese contents be maintained between 1.0 and 2.0%, and ideally at about 1.5%. Obviously, carbon contents should not be too low, e.g. below about 0.03%, as carbon is essential for formation of the carbide precipitates. In addition, it is well known that the amount of columbium in solution within the austenite will strongly effect the  $A_{r3}$  transformation temperature. Since columbium carbides or carbonitrides will precipitate during hot rolling above the  $A_{r3}$  transition temperature, the amount of columbium remaining in solution at transformation may be uncertain. In addition, it is known that decreasing the

finishing temperature and/or increasing the amount of deformation will raise the  $A_{r3}$  transformation temperature somewhat. Therefore, although the composition limits and hot rolling parameters disclosed above will be sufficient to realize the advantages of this invention, for optimum results with any given alloy composition and given hot rolling schedule, it is preferable that the  $A_{r3}$  transformation temperature be determined under actual rolling conditions. It should also be noted that high levels of all three constituents, manganese, molybdenum and columbium, and especially high levels of manganese, should be avoided to assure transformation to polygonal ferrite rather than acicular ferrite, because the  $A_{r3}$  temperature of acicular ferrite is about 1250°F or less, which is below the desired range described above.

In order to optimize both strength and toughness in this steel the use of vanadium should be avoided. However, in applications where toughness is not critical, up to 0.20% vanadium can be added to the steel to ordinarily increase yield strength by up to 10 ksi for an addition of 0.08%, or up to 15 ksi for an addition of 0.20% vanadium. It should be understood however that while vanadium will increase yield strength, it will impair the impact transition temperature consistent with known effects of precipitation strengthening.

A further unexpected feature of this invention steel is that tempering the steel, subsequent to the hot-rolling, will in some cases increase the yield strength by about 10 ksi with no or very little impairment of the impact properties. Those skilled in the art would normally think that the relatively high  $A_{r3}$  transition temperature, as characteristic of these steels, would cause complete precipitation strengthening of the steel in the as-hot-worked condition, and that no secondary hardening response would be expected. On the other hand, secondary hardening of such magnitude in other steels is normally accompanied by a 50° to 70°F increase (impairment) in impact-transition temperature. This is not observed however in the steel of this invention. The tempering response of the steels can be very rapid, but over-aging is slow so that a wide variety of tempering time and temperature, times from one minute to two hours and temperatures within a range of about 1100° to 1250°F can be used to attain uniform mechanical properties without risk of over-aging. Although this mechanism is not completely understood I believe this secondary hardening results not from precipitation strengthening but from relief of associated residual microstresses.

As noted above, this unusual tempering response is noted only in some cases. Specifically, it can be realized only with those alloys wherein the manganese and/or molybdenum are on the higher side of the recited range. Although these limits are not well defined, the tempering improvement will not be effective on alloys having less than 1.20% manganese and less than 0.20% molybdenum, and will be effective if manganese contents exceed about 1.30% and/or molybdenum contents exceed about 0.25%. In the area therebetween, further strengthening with a tempering treatment may or may not be effective depending upon the combined amounts of manganese and molybdenum.

Although those steels with relatively low manganese and molybdenum contents exhibit essentially no strengthening response when tempered, these steels are somewhat stronger than the higher manganese and/or

molybdenum steels in the hot-rolled condition. This characteristic makes these lower manganese and molybdenum steels more ideally suited to applications where the added cost or inconvenience of tempering cannot be tolerated.

In view of the above considerations, an optimum composition for the steel of this invention to be used in the as-hot-rolled condition is 0.05 to 0.10% carbon, 1.0 to 1.3% manganese, 0.15 to 0.25% molybdenum and 0.02 to 0.05% columbium. If this composition is hot rolled at a temperature above 1500°F, cooled to about 1400°F and then hot rolled again at this intercritical temperature to effect a thickness reduction of 20 to 30%, yield strengths of about 75 ksi can be achieved in combination with excellent notch toughness, i.e. with FATT values of about -80°F. On the other hand, an optimum composition of the steel of this invention that may be strengthened by tempering would differ from the above composition in requiring 1.3 to 1.6% manganese and 0.20 to 0.40% molybdenum. If this latter composition is hot rolled at temperatures above 1500°F, cooled to about 1400°F and then hot rolled at this intercritical temperature to effect a thickness reduction of 20 to 30%, yield strengths of about 70 ksi can be achieved in the as-hot-rolled condition, and about 80 ksi in the tempered condition, in either case, combined with excellent notch toughness, i.e. FATT values of about -80°F.

The combination of low carbon and moderate manganese contents of the inventive steels ensures good weldability. Although it might be thought that welding could have a very adverse effect on the base plate in the region of the heat affected zone (HAZ) and deleteriously affect the mechanical properties established by the intercritical rolling, test have shown that such is not the case. For example, for large diameter submerged-arc-welded line pipe made from the steels of this invention, standard transverse strip tensile tests across the weld and standard transverse Charpy V-notch impact tests with the notch located in the HAZ region, have shown that the strength and impact properties of the HAZ are similar to those of the pipe body.

Another unexpected and beneficial characteristic of the steel of this invention can be realized when the steel is made into line-pipe using the conventional "U and O" process. Normally, when using steel plate with compositions and rolling histories ordinarily associated with line-pipe grades, the yield strength of the line-pipe tested after forming and welding is much lower than that of the original plate (because of the well-known Bauschinger effect). Therefore, such line-pipe is cold-expanded (increased in diameter) by up to about 2% to strain harden the steel to increase the yield strength to a value approximately equal to that of the original plate. Even in such an expanded condition, prior art line-pipe frequently exhibits yield strengths below that of the original plate.

Although cold expansion is generally desirable to obtain consistent dimensional tolerances (diameter and roundness), the degree of cold expansion should not be excessive because such plastic straining detracts from the overall ductility of the steel. In addition, the unreliable changes in yield strength for prior art line-pipe grades, as noted above, make it very difficult if not at times impossible, to estimate the final line-pipe yield strength knowing only the original plate strength. Therefore, plates which may result in line-pipe having

unacceptable yield strengths cannot always be sorted out prior to the actual making and testing of the pipe. This, of course, can result in a rather costly procedure.

To my surprise, steel plates of the present invention, when made into pipe using the U and O process may exhibit an increase in yield strength in the unexpanded condition, and may exhibit pronounced further strengthening when expanded. Hence, the final pipe strength is essentially equal to or considerably greater than that of the original plate. Those steels which exhibit the largest increases in yield strength are those rolled steels containing the higher manganese and/or molybdenum contents, i.e. those responsive to tempering, and the increase in yield strengths achieved by pipe forming correspond closely with yield strength increases achieved by tempering. It appears therefore that the two strengthening mechanisms are closely related, and I believe that both are due to elimination of residual stresses in the as-hot-rolled plates.

Accordingly, plates which have compositions which would not normally show an increase in yield strength if tempered, and plates which have been tempered, will, after fabrication into pipe by the U and O process and expanded, exhibit yield strengths that remain substantially constant or increase only slightly. On the other hand, if those steel plates having compositions which are responsive to tempering, are fabricated into line-pipe by the U and O process and expanded will exhibit an increase in yield strength thereby eliminating any need for tempering the steel before or after pipe fabrication.

### EXAMPLES

In one test, a number of steels were made as 100-pound air-induction-melted heats and cast as slab ingots. These slabs were heated to about 2250° to 2300°F, and then hot-rolled in fourteen reduction passes to ½-inch thick plates with the first pass at about 2200°F, the last pass either at 1540°F or at 1400°F, and the remaining passes distributed more or less uniformly

over the temperature ranges 2200° to 1540°F or 2200° to 1400°F. By drilling a hole in each slab, and inserting a thermocouple in each slab prior to rolling, the pass temperatures could be recorded, and the start of the austenite-to-ferrite transformation could be detected by the obvious change in the rate of cooling (thermal arrest) produced by the heat of transformation.

Table I gives the chemical compositions and Table II the mechanical properties of these steels. Some of the steels were finish-rolled at 1540°F which is above their upper-critical-transformation temperature, and the others were finish-rolled at 1400°F, which for all of these steels is below their upper-critical temperature. The microstructures of the steels showed the characteristic veined and deformed ferrite grains with the volume fraction of this deformed ferrite varying with alloy content in a manner consistent with the effects of carbon, manganese, molybdenum, columbium, and vanadium on changing the upper-critical transformation temperature. All of the steel plates finish-rolled at 1400°F received from 11.5 to 31.5 percent reduction in thickness below their upper-critical transformation temperatures which varied from about 1425° to 1500°F.

TABLE I

| Steel* | CHEMICAL COMPOSITIONS |      |      |       | V      |
|--------|-----------------------|------|------|-------|--------|
|        | C                     | Mn   | Mo   | Cb    |        |
| D      | 0.067                 | 1.22 | 0.18 | 0.035 | —      |
| E      | 0.064                 | 1.25 | 0.14 | 0.028 | —      |
| F      | 0.069                 | 1.25 | 0.31 | 0.037 | —      |
| G      | 0.075                 | 1.14 | 0.27 | 0.034 | —      |
| H      | 0.072                 | 1.13 | 0.18 | 0.035 | 0.071  |
| I      | 0.076                 | 1.10 | 0.14 | 0.030 | 0.085  |
| J      | 0.070                 | 1.10 | 0.32 | 0.035 | 0.071  |
| K      | 0.079                 | 1.14 | 0.27 | 0.034 | 0.081  |
| L      | 0.075                 | 1.38 | 0.25 | 0.034 | <0.005 |
| M      | 0.079                 | 1.43 | 0.25 | 0.038 | 0.091  |
| N      | 0.087                 | 0.91 | 0.37 | 0.035 | 0.089  |
| O      | 0.080                 | 1.23 | 0.32 | 0.033 | 0.084  |
| P      | 0.069                 | 1.40 | 0.34 | 0.028 | 0.078  |
| Q      | 0.080                 | 1.43 | 0.26 | <0.01 | 0.091  |

\*All steels were Si-Al-killed and contained about 0.010% P, from 0.011 to 0.022% S, and about 0.004 to 0.007% N. Compositions are in weight percent.

TABLE II

MECHANICAL PROPERTIES OF HOT-ROLLED AND OF TEMPERED ½ INCH THICK PLATES

| Steel | Finishing Temp., F | Condition ** | Yield Strength (0.2% Offset), ksi | Tensile Strength ksi | CVN 50% Shear Fracture Appearance Transition Temp., F |
|-------|--------------------|--------------|-----------------------------------|----------------------|---|
| D     | 1540               | HR           | 62.5                              | 73.3                 | -70   |
|       | 1540               | HR + T       | 62.8                              | 72.3                 | -90   |
| E     | 1400               | HR           | 70.0                              | 78.5                 | —   |
|       | 1400               | HR + T       | 71.7                              | 77.8                 | -70   |
| F     | 1540               | HR           | 59.1                              | 76.5                 | -85   |
|       | 1540               | HR + T       | 66.5                              | 75.0                 | -85   |
| G     | 1400               | HR           | 67.7                              | 83.2                 | —   |
|       | 1400               | HR + T       | 76.3                              | 83.5                 | -75   |
| H     | 1540               | HR           | 66.6                              | 75.1                 | -50   |
|       | 1540               | HR + T       | 65.2                              | 74.9                 | -45   |
| I     | 1400               | HR           | 75.6                              | 85.1                 | —   |
|       | 1400               | HR + T       | 73.6                              | 81.9                 | -45   |
| J     | 1540               | HR           | 65.3                              | 76.4                 | -65   |
|       | 1540               | HR + T       | 67.6                              | 77.1                 | -75   |
| K     | 1400               | HR           | 78.0                              | 92.9                 | —   |
|       | 1400               | HR + T       | 81.2                              | 88.9                 | -50   |
| L     | 1400               | HR           | 67.5                              | 84.7                 | —   |
|       | 1400               | HR + T       | 75.3                              | 81.2                 | -80   |
| M     | 1400               | HR           | 69.7                              | 90.7                 | —   |
|       | 1400               | HR + T       | 84.0                              | 91.7                 | -50   |
| N     | 1400               | HR           | 75.8                              | 90.8                 | —   |
|       | 1400               | HR + T       | 87.3                              | 95.6                 | -20   |

TABLE II--Continued

MECHANICAL PROPERTIES OF HOT-ROLLED AND OF TEMPERED 1/2 INCH THICK PLATES

| Steel | Finishing Temp., F | Condition ** | Yield Strength (0.2% Offset), ksi | Tensile Strength ksi | CVN 50% Shear Fracture Appearance Transition Temp., F |
|-------|--------------------|--------------|-----------------------------------|----------------------|---|
| O     | 1400               | HR           | 70.9                              | 88.5                 | —   |
|       | 1400               | HR + T       | 88.7                              | 94.0                 | -50   |
| P     | 1400               | HR           | 64.5                              | 85.6                 | -80   |
|       | 1400               | HR + T       | 89.3                              | 91.9                 | -45   |
| Q     | 1400               | HR           | 60.5                              | 82.9                 | —   |
|       | 1400               | HR + T       | 71.8                              | 80.7                 | -55   |

\*\*HR denotes the hot-rolled condition, and HR + T denotes the hot-rolled plus tempered (1 hour at 1200°F) condition.

Table III below exemplifies the increase in yield strength which can be achieved by forming steel plates of this invention into line-pipe using the conventional U and O process and expanding. Steels 1 and 2 are conventional prior art line-pipe steels, while steels 3-12 are steels according to this invention. The steels were fabricated into 30, 36 or 42 inch diameter line-pipe.

- ture;
- c. hot rolling said heated slab at a temperature above the Ar<sub>3</sub> transition temperature sufficient to effect no more than 90% of the intended hot reduction;
- d. cooling the partially hot-rolled slab to a temperature below the Ar<sub>3</sub> transition temperature but above the Ar<sub>1</sub> transition temperature to cause a

TABLE III

| Steel | Chemical Composition*, wt. %                   |      |      |       |      | Plate Gage Inch | Plate Strength, ksi |         | Unexpanded Pipe Strength, ksi |         | Expanded (~1.5%) Pipe Strength, ksi |         |
|-------|--|------|------|-------|------|-----------------|---------------------|---------|-------------------------------|---------|-------------------------------------|---------|
|       | C  | Mn   | Mo   | Cb    | V    |                 | Yield               | Tensile | Yield                         | Tensile | Yield                               | Tensile |
|       |  |      |      |       |      |                 |                     |         |                               |         |                                     |         |
| 1     | 0.14   | 1.34 | —    | 0.035 | 0.06 | 0.462           | 71.2                | 88.5    | 68.0                          | 88.0    | 72.5                                | 89.4    |
| 2++   | 0.14   | 1.34 | —    | 0.035 | 0.06 | 0.462           | 74.5                | 88.4    | 68.3                          | 89.5    | 69.1                                | 89.9    |
|       | 0.14   | 1.36 | —    | —     | 0.08 | 0.424           | 68.3                | 87.4    | 59.8                          | 87.7    | 73.6                                | 91.2    |
|       | 0.14   | 1.36 | —    | —     | 0.08 | 0.424           | 70.1                | 87.8    | 65.3                          | 87.4    | 71.7                                | 90.8    |
|       |  |      |      |       |      |                 |                     |         |                               |         |                                     |         |
| 3     | 0.08   | 1.42 | 0.29 | 0.033 | —    | 0.515           | 71.5                | 92.8    | —                             | —       | 83.7                                | 97.5    |
| 4     | 0.08   | 1.41 | 0.16 | 0.032 | —    | 0.515           | 63.4                | 92.8    | —                             | —       | 80.0                                | 98.7    |
| 5     | 0.08   | 1.15 | 0.17 | 0.044 | —    | 0.515           | 75.2                | 85.2    | —                             | —       | 82.6                                | 95.5    |
| 6     | 0.07   | 1.15 | 0.17 | 0.054 | —    | 0.515           | 80.5                | 89.7    | —                             | —       | 83.8                                | 92.6    |
| 7     | 0.08   | 1.29 | 0.18 | 0.05  | —    | 0.312           | 74.2                | 85.7    | —                             | —       | 77.1                                | 91.9    |
|       | 0.08   | 1.29 | 0.18 | 0.05  | —    | 0.500           | 75.0                | 85.1    | —                             | —       | 75.0                                | 92.5    |
| 8     | 0.07   | 1.24 | 0.19 | 0.04  | —    | 0.312           | 71.6                | 87.6    | —                             | —       | 78.0                                | 92.0    |
|       | 0.07   | 1.24 | 0.19 | 0.04  | —    | 0.500           | 64.9                | 81.1    | —                             | —       | 68.2                                | 84.7    |
|       | 0.07   | 1.24 | 0.19 | 0.04  | —    | 0.625           | 65.2                | 76.4    | —                             | —       | 64.3                                | 79.3    |
| 9     | 0.10   | 1.24 | 0.19 | 0.05  | —    | 0.688           | 67.9                | 80.2    | 64.1                          | 84.5    | 70.0                                | 86.9    |
| 10    | 0.10   | 1.11 | 0.20 | 0.05  | —    | 0.875           | 65.4                | 78.1    | 61.7                          | 81.2    | 68.6                                | 83.3    |
| 11    | 0.13   | 1.30 | 0.28 | 0.06  | —    | 1.00            | 68.0                | 85.5    | 81.8                          | 94.4    | 80.6                                | 93.9    |
| 12    | Tempered (1.5 hours at 1100°F) Mn-Mo-Cb Plates |      |      |       |      |                 |                     |         |                               |         |                                     |         |
|       | 0.06   | 1.24 | 0.31 | 0.06  | —    | 0.500           | 79.6                | 84.3    | —                             | —       | 81.2                                | 94.2    |
|       | 0.06   | 1.24 | 0.31 | 0.06  | —    | 0.800           | 80.3                | 86.5    | —                             | —       | 81.1                                | 90.9    |
|       | 0.06   | 1.24 | 0.31 | 0.06  | —    | 0.800           | 75.4                | 81.2    | 75.7                          | 85.7    | 74.7                                | 88.8    |
|       | 0.06   | 1.24 | 0.31 | 0.06  | —    | 1.00            | 74.0                | 83.3    | 75.9                          | 94.5    | 75.4                                | 91.9    |

Notes for Table III:

\* Determined by strip tension specimens.

+ All steels were Si-Al-killed.

\*\* Steel 2 also contained 0.18% Cu, 0.18% Cr and 0.08% Ni.

I claim:

1. A process for producing a hot-rolled high-strength low-alloy steel having a yield strength of at least 65 ksi and exceptional toughness at subzero temperatures characterized by 50% shear FATT values at temperatures as low as -80°F, the steps comprising:

a. forming a steel slab having the following composition by weight:

|                     |               |
|---------------------|---------------|
| carbon              | 0.03 to 0.15% |
| phosphorus          | 0.04 % max.   |
| sulfur              | 0.04 % max.   |
| manganese           | 0.5 to 2.0 %  |
| molybdenum          | 0.1 to 0.40%  |
| columbium           | 0.01 to 0.10% |
| vanadium            | 0 to 0.20%    |
| iron and impurities | balance;      |

b. heating said slab to a temperature sufficiently above the Ar<sub>3</sub> transition temperature to austenitize the microstructure and dissolve all carbide and nitride precipitates into the austenitic microstruc-

portion of the austenitic microstructure to transform to ferrite;

e. further hot rolling said partially hot rolled slab at a temperature between the Ar<sub>3</sub> and Ar<sub>1</sub> transition temperature sufficient to effect a thickness reduction of from 10 to 40%; and

f. thereafter cooling the hot rolled steel to ambient temperatures whereat said steel is characterized by the presence of both equiaxed and cold-worked ferrite grains with a uniform distribution of carbide and nitride precipitates throughout.

2. The process of claim 1 in which the steel slab formed has the following composition by weight:

|                     |               |
|---------------------|---------------|
| carbon              | 0.05 to 0.10% |
| phosphorus          | 0.04 % max.   |
| sulfur              | 0.04 % max.   |
| manganese           | 1.3 to 1.6 %  |
| molybdenum          | 0.20 to 0.40% |
| columbium           | 0.02 to 0.05% |
| vanadium            | nil           |
| iron and impurities | balance       |

3. The process of claim 1 in which the steel slab formed has the following composition by weight:

|                     |               |
|---------------------|---------------|
| carbon              | 0.05 to 0.10% |
| phosphorus          | 0.04 % max.   |
| sulfur              | 0.04 % max.   |
| manganese           | 1.0 to 1.3 %  |
| molybdenum          | 0.15 to 0.25% |
| columbium           | 0.02 to 0.05% |
| vanadium            | nil           |
| iron and impurities | balance       |

4. The process of claim 1 in which the steel slab formed further contains small additions of strengthening elements selected from the group consisting of nickel, copper and chromium which are added as strengtheners and to impart corrosion resistance.

5. The process of claim 1 in which the steel slab is heated to a temperature above 2000°F prior to hot rolling to assure dissolution of all carbide and nitride precipitates, and thereafter performing the step (c) hot rolled at a temperature within the range 1500°F to said temperature above 2000°F.

6. The process of claim 1 in which the step (e) hot rolling between the Ar<sub>3</sub> and Ar<sub>1</sub> transition temperatures is sufficient to effect a thickness reduction of from 20 to 30%.

7. The process of claim 1 in which the partially hot rolled steel as cooled at step (d) is cooled to a temperature at least 25°F below the Ar<sub>3</sub> transition temperature.

8. The process of claim 1 in which the partially hot rolled steel as cooled at step (d) is cooled to a temperature just slightly above the Ar<sub>1</sub> transition temperature.

9. The process of claim 2 in which the finished hot rolled steel is tempered at a temperature within the range 1100° to 1250°F for a period of 1 to 120 minutes at temperature to increase the hot-rolled steel's yield strength by about 10 ksi with little impairment of the steel's impact properties.

10. A ferritic hot-rolled high-strength low-alloy steel having a yield strength of at least 65 ksi and exceptional toughness at subzero temperatures characterized by 50% shear FATT values at temperatures as low as -80°F, said steel consisting essentially of the following composition by weight:

|                     |               |
|---------------------|---------------|
| carbon              | 0.03 to 0.15% |
| phosphorus          | 0.04 % max.   |
| sulfur              | 0.04 % max.   |
| manganese           | 0.5 to 2.0 %  |
| molybdenum          | 0.1 to 0.40%  |
| columbium           | 0.01 to 0.10% |
| vanadium            | 0 to 0.20%    |
| iron and impurities | balance       |

and having a microstructure containing both equiaxed and cold-worked polygonal ferrite grains and a uniform distribution of carbide and nitride precipitates throughout, said precipitates serving to strengthen the steel through precipitation hardening effects, grain refining effects and by pinning and stabilizing the cold-worked ferrite grains to retain a high dislocation density therein and the strengthening effects thereof.

11. A ferritic hot-rolled high-strength low-alloy steel according to claim 10 consisting essentially of the following composition by weight:

|            |               |
|------------|---------------|
| carbon     | 0.05 to 0.10% |
| phosphorus | 0.04 % max.   |
| sulfur     | 0.04 % max.   |

-Continued

|                     |               |
|---------------------|---------------|
| manganese           | 1.3 to 1.6 %  |
| molybdenum          | 0.20 to 0.40% |
| columbium           | 0.02 to 0.05% |
| vanadium            | nil           |
| iron and impurities | balance       |

12. A ferritic hot-rolled high-strength low-alloy steel according to claim 10 consisting essentially of the following composition by weight:

|                     |               |
|---------------------|---------------|
| carbon              | 0.05 to 0.10% |
| phosphorus          | 0.04 % max.   |
| sulfur              | 0.04 % max.   |
| manganese           | 1.0 to 1.3 %  |
| molybdenum          | 0.15 to 0.25% |
| columbium           | 0.02 to 0.05% |
| vanadium            | nil           |
| iron and impurities | balance       |

13. A process for producing high-strength, low-alloy steel line-pipe having a yield strength of at least 65 ksi and exceptional toughness at subzero temperatures characterized by 50% shear FATT values at temperatures as low as -80°F, the steps comprising:

a. forming a steel slab having the following composition by weight:

|                     |               |
|---------------------|---------------|
| carbon              | 0.03 to 0.15% |
| phosphorus          | 0.04 % max.   |
| sulfur              | 0.04 % max.   |
| manganese           | 0.5 to 2.0 %  |
| molybdenum          | 0.1 to 0.40%  |
| columbium           | 0.01 to 0.10% |
| vanadium            | 0 to 0.20%    |
| iron and impurities | balance;      |

b. heating said slab to a temperature sufficiently above the Ar<sub>3</sub> transition temperature to austenitize the microstructure and dissolve all carbide and nitride precipitates into the austenitic microstructure;

c. hot rolling said heated slab at a temperature above the Ar<sub>3</sub> transition temperature sufficient to effect no more than 90% of the intended hot reduction;

d. cooling the partially hot-rolled slab to a temperature below the Ar<sub>3</sub> transition temperature but above the Ar<sub>1</sub> transition temperature to cause a portion of the austenitic microstructure to transform to ferrite;

e. further hot rolling said partially hot rolled slab at a temperature between the Ar<sub>3</sub> and Ar<sub>1</sub> transition temperature sufficient to effect a thickness reduction of from 10 to 40%;

f. thereafter cooling the hot rolled steel to ambient temperatures whereat said steel is characterized by a yield strength of at least 65 ksi; and

g. fabricating the hot rolled steel into line-pipe using the conventional U and O process, whereupon said line-pipe is characterized by a yield strength of at least 65 ksi.

14. A process according to claim 13 wherein said steel slab contains 1.3 to 1.6 wt. % manganese and 0.20 to 0.40 wt. % molybdenum, and said line-pipe is characterized by a yield strength of at least 70 ksi.

15. A high-strength, low-alloy steel line-pipe having a yield strength of at least 65 ksi and exceptional toughness at subzero temperatures characterized by 50% shear FATT values at temperatures as low as -80°F consisting essentially of:

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|                     |               |
|---------------------|---------------|
| carbon              | 0.03 to 0.15% |
| PHOSPHORUS          | 0.04 % max.   |
| sulfur              | 0.04 % max.   |
| manganese           | 0.5 to 2.0 %  |
| molybdenum          | 0.1 to 0.40%  |
| columbium           | 0.01 to 0.10% |
| vanadium            | 0 to 0.20%    |
| iron and impurities | balance       |

and a microstructure containing both equiaxed and cold-worked polygonal ferrite grains and a uniform dis-

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tribution of carbide and nitride precipitates throughout, said cold-worked ferrite grains having a high dislocation density which is pinned and stabilized by the carbide and nitride precipitates.

16. A line-pipe according to claim 15 in which said manganese is within range 1.3 to 1.6, said molybdenum is within the range 0.20 to 0.40% and said line-pipe is characterized by a yield strength of at least 70 ksi.

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