United States Patent

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[72] Inventors **Koichi Aoki**
Yokohama;
Seinosuke Yano, Kamakura; Hiroshi Mimura, Tokyo; Tohru Inoue, Tokyo; Hiroshi Sakurai, Kawasaki, all of Japan

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[73] Assignee **Yawata Iron & Steel Co., Ltd.**
Tokyo, Japan

[54] **METHOD OF HEAT-TREATING LOW TEMPERATURE TOUGH STEEL**
8 Claims, 12 Drawing Figs.

[52] **U.S. Cl.** **148/12,** 148/12.4, 148/134, 148/143
[51] **Int. Cl.** **C21d 1/26,** C21d 7/14
[50] **Field of Search** 148/12, 12.4, 134, 143; 75/123

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Primary Examiner—L. Dewayne Rutledge
Assistant Examiner—W. W. Stallard
Attorney—Wenderoth, Lind & Ponack

ABSTRACT: Heat-treating method of hot-rolled steel plate containing 1.5% to 10.0% Ni, 0.1% to 5.0% Mn and further at least one element selected from the group consisting of Mo, Cu, Cr, Nb and V, at need and the balance being substantially Fe, comprising subjecting one or more times repeatedly said hot-rolled steel plate to heating to and sequent quenching from a temperature within the range between Ac_1 and Ac_3 points, then subjecting the steel plate to tempering treatment at a temperature below Ac_1 point, thereby to improve largely toughness at low temperature and obtain excellent workability.

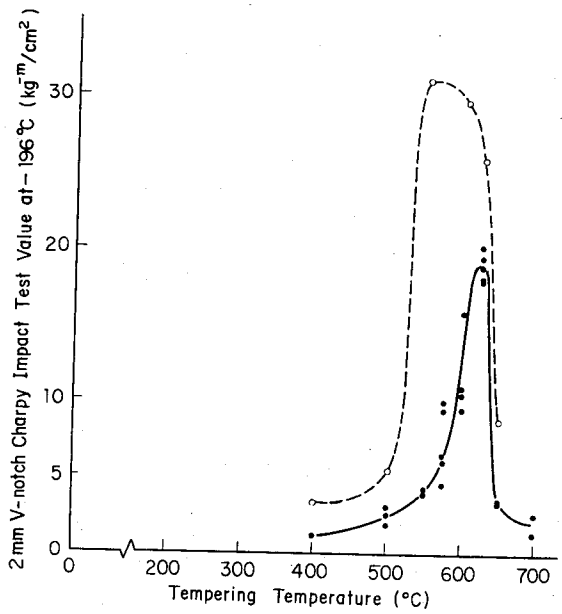
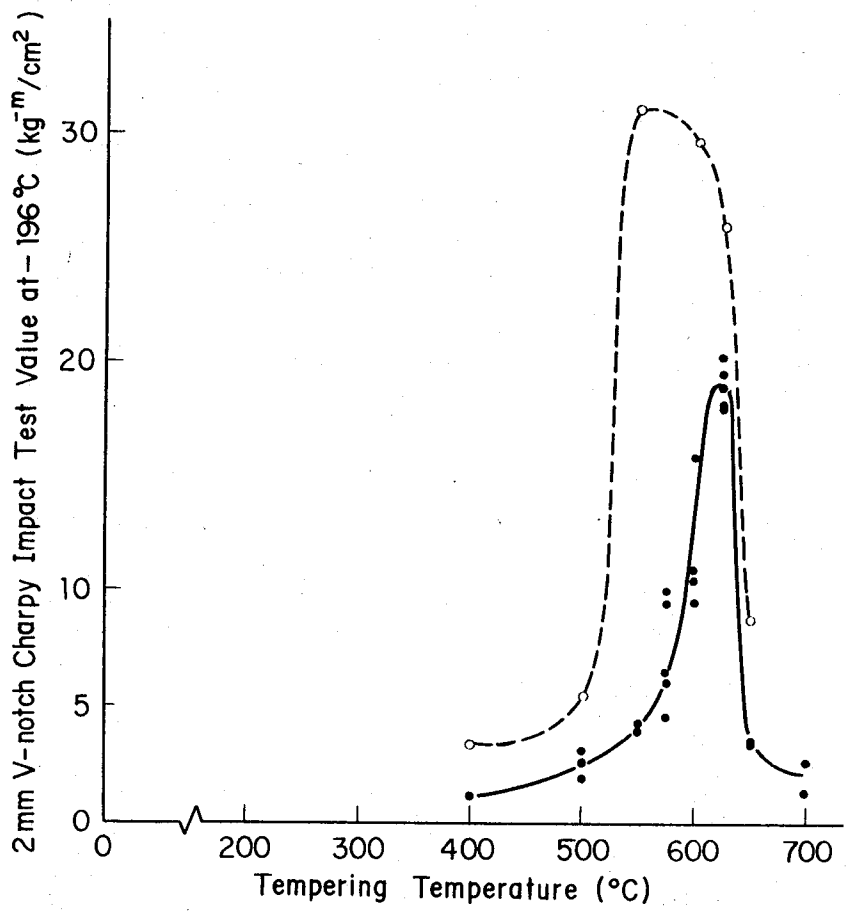


FIG. 1



INVENTORS

Koichi Aoki
Seinosuke Yano
Hiroshi Mimura
Tohru Inoue
Hiroshi Sakurai

BY, Wenderoth, Lind & Ponack
ATTORNEYS

FIG. 3

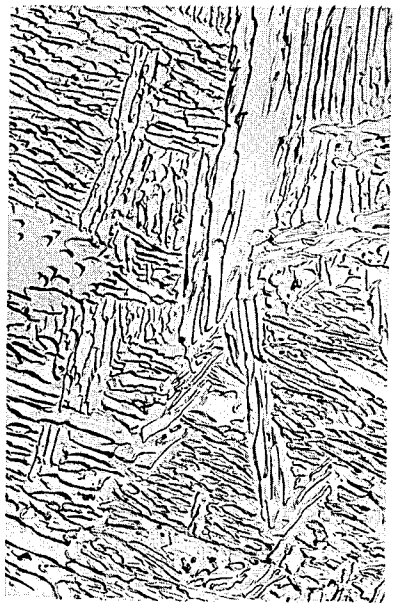


FIG. 5



FIG. 2

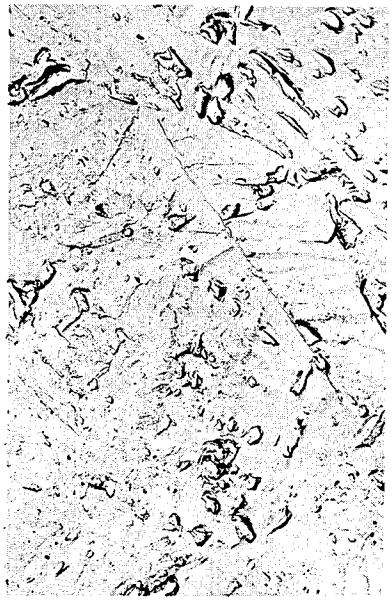
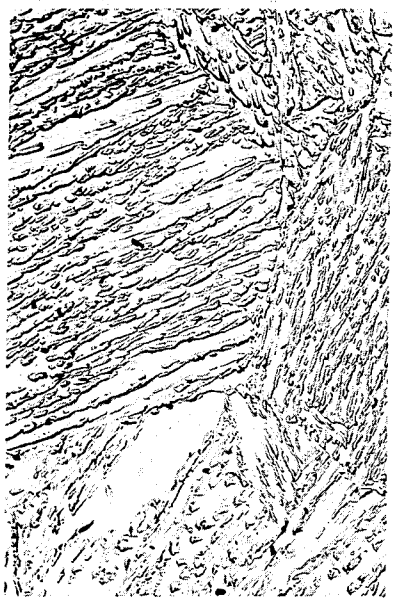


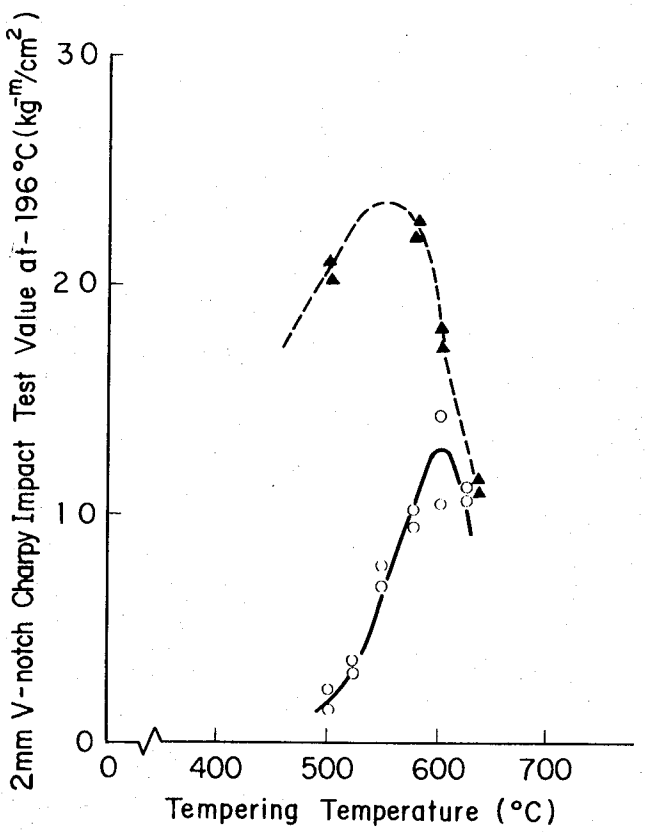
FIG. 4



INVENTORS
KOICHI AOKI
SEINOSUKE YANO
HIROSHI MIMURA
TOHRU INOUE
HIROSHI SAKURAI

BY
Wenderoth, Lind & Ponack
ATTORNEYS

FIG. 6



INVENTORS
Koichi Aoki
Seinosuke Yano
Hiroshi Mimura
Tohru Inoue
Hiroshi Sakurai

BY Wenderoth, Lind & Ponack
ATTORNEYS

FIG. 8

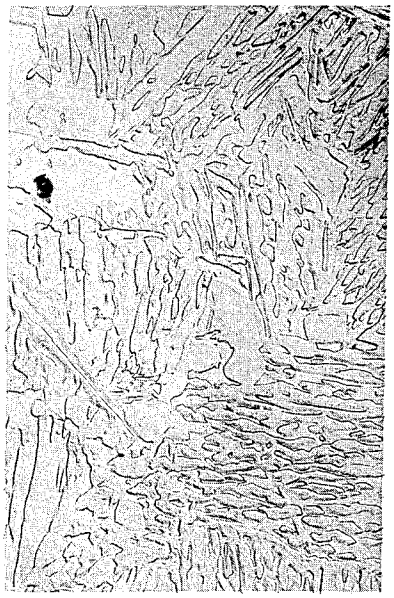


FIG. 9

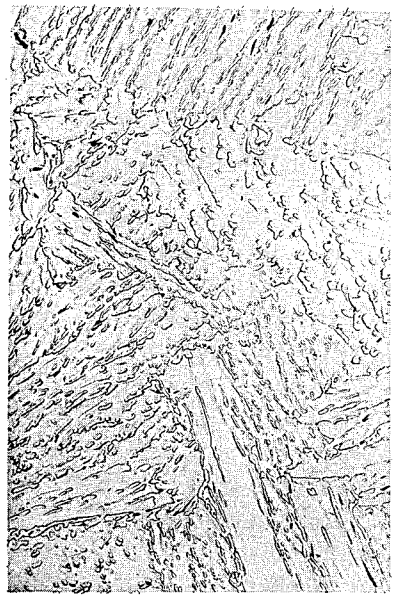


FIG. 7

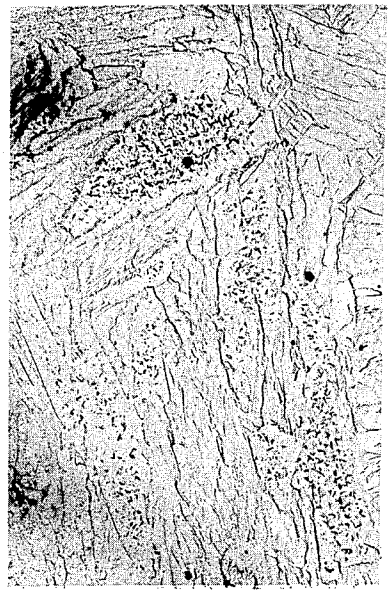
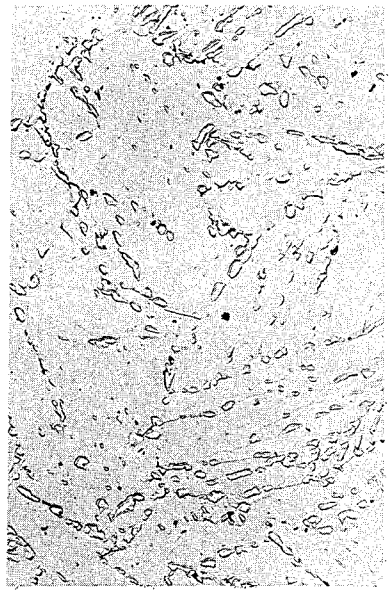


FIG. 10



INVENTORS
KOICHI AOKI
SEINOSUKE YANO
HIROSHI MIMURA
TOHRU INOUE
HIROSHI SAKURAI

BY
Wenderoth, Lind & Ponack
ATTORNEYS

FIG.12

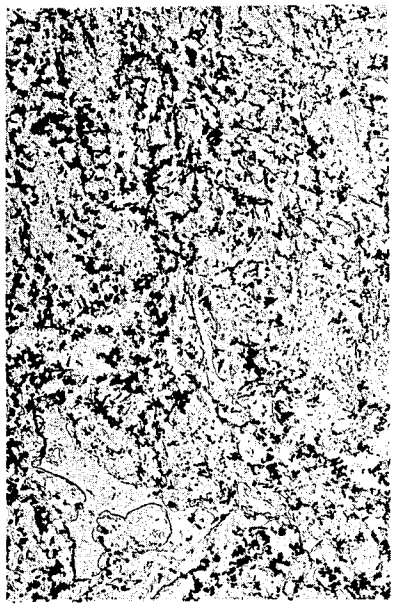
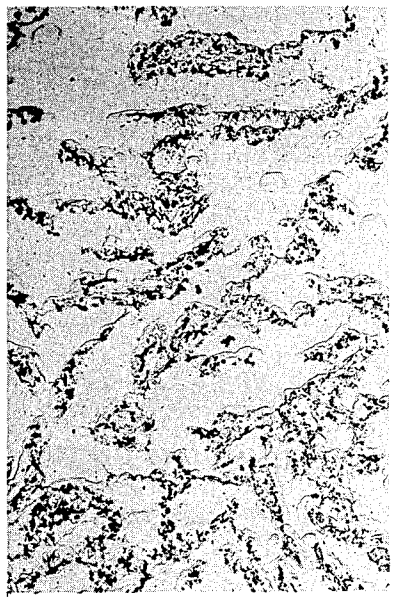


FIG.11



INVENTORS
KOICHI AOKI
SEINOSUKE YANO
HIROSHI MIMURA
TOHRU INOUE
HIROSHI SAKURAI

BY
Wenderoth, Lind & Ponack
ATTORNEYS

METHOD OF HEAT-TREATING LOW TEMPERATURE TOUGH STEEL

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to a heat-treating method for imparting a very high toughness at low temperature, particularly at such an extremely low temperature as −196° C., the boiling point of liquid nitrogen, and an excellent workability, particularly low yield ratio at room temperature to a low carbon tough steel containing 1.5 to 10.0 wt.% Ni and 0.1 to 5.0 wt.% Mn.

2. Description of the Prior Art

It is well known that recently a 9%-Ni steel is widely used as a steel material for use at low temperature. This 9%-Ni steel is usually made a finished product after subjected to hardening and tempering treatments or to normalizing and tempering treatments after a solid solution treatment. The thus treated product shows a relatively good toughness at low temperature, but it does not always follow that a satisfactory result can be obtained in case an optimum toughness superior to the above-mentioned is required.

As regards to workability, no particularly excellent result could be obtained according to the experiments made by the inventors of the present invention.

Further a 6%-Ni series low temperature tough steel has been developed by the inventors of the present invention as a low temperature tough steel which should take the place of the 9%-Ni steel, and for this new steel U.S. Pat. No. 3,388,988 was granted. By subsequent investigations, however, it was discovered that in order to obtain an optimum toughness a tempering treatment must be carried out under the optimum conditions of a relatively narrow range, which make a knotty point in the operation. Also as regards the workability, the optimal result could not still be secured with the 6%-Ni steel as in the case of the 9%-Ni steel.

Now, as seen from examples of 6%-Ni and 9-Ni steels, in general, with a usual heat treatment which is to be carried out for obtaining a high toughness the yield ratio becomes high, but a steel having a high yield ratio is used to be detested by a construction designer from the view point of safety. As is explained in the following, the present invention is to provide a heat-treating method, by which a steel having a high toughness can be obtained in the wide range of a tempering temperature, while keeping the yield ratio low by utilizing a segregation.

SUMMARY OF THE INVENTION

The present invention is to eliminate the above-mentioned defects of Ni-containing steels. Therefore, the primary object of the present invention is to provide an excellent tough steel which is by far improved in the toughness by causing an Ni-containing steel to form a fine structure according to a method mentioned in the following, without causing thereby a reduction in a strength at low temperature.

The second object of the present invention is to impart an excellent workability, particularly a property of low yield ratio to an Ni-containing tough steel.

Another object of the present invention is to provide a tempering treatment of an Ni-containing steel with the optimum conditions of a wider range.

Still other objects of the present invention will become manifest by the following descriptions with reference to the examples and accompanying drawings.

The present invention which is to attain the above-mentioned objects provides the method, wherein a hot-rolled steel plate containing 1.5 to 10.0% Ni, 0.1 to 5.0% Mn, and Si in an amount as required for making steel (preferably 0.05 to 0.4%) and further at least one element properly selected from the group consisting of 0.05 to 1.0% Mo (a part or the whole of Mo may be replaced by W), 0.10 to 2.0% Cu, 0.10 to 1.50% Cr, less than 0.05% sol. Al, less than 0.2% Nb, less than 0.2% V, less than 0.1% Zr, less than 0.1% Ti, less than 0.005% B, as occasion demand, and the balance being Fe and unavoidable impurities is heated to a temperature above the Ac_3 transformation point and then cooled with a cooling rate greater than a cooling by air and thereafter is heated to a temperature between the Ac_1 transformation point and the Ac_3 transformation point and then cooled (with a cooling rate greater than a cooling by air) and then the thus treated steel plate is further subjected to a tempering treatment at a temperature below the Ac_1 transformation point, or the hot-rolled steel material may be directly heated to and quenched from a temperature between the Ac_1 transformation point and the Ac_3 transformation point, and then the thus treated steel plate is subjected to a tempering treatment at temperature below the Ac_1 transformation point. Further, prior to the heating and cooling to and from a temperature above the Ac_3 transformation point or to and from a temperature between the Ac_1 transformation point and the Ac_3 transformation point a solid solution treatment may be carried out.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a diagram showing the relation between a tempering temperature and a toughness of a 6%-Ni series steel treated according to the method of the present invention as compared with that of a 6%-Ni series steel treated according to a conventional method, i.e., quenched and tempered.

FIG. 2 is a photograph showing a structure by the observation with an electron microscope of a magnification of 4,500 of a 6%-Ni series hot-rolled steel plate as heated to 800° C. (above the Ac_3 transformation point) and cooled therefrom.

FIG. 3 is a photograph showing a structure by the observation with an electron microscope of a magnification of 4,500 of in the case where the steel of FIG. 2 is further heated and sequently cooled to and from 670° C.

FIG. 4 is a photograph showing a structure by the observation with an electron microscope of a magnification of 4,500 of in the case where the steel plate of FIG. 3 is further subjected to a tempering treatment at 600° C.

FIG. 5 is a photograph showing a structure by the observation with an electron microscope of a magnification of 4,500 of in the case where the steel plate of FIG. 2 is directly subjected to a tempering treatment at 600° C.

FIG. 6 is a diagram showing the relation between a tempering temperature and a toughness of a 9%-Ni series steel treated according to the method of the present invention as compared with that of a 9%-Ni series steel treated according to a conventional method.

FIG. 7 is a photograph showing a structure by the observation with an electron microscope of a magnification of 4,500 of a 9%-Ni series hot-rolled steel plate as heated to 800° C. and cooled therefrom.

FIG. 8 is a photograph showing a structure by the observation with an electron microscope of a magnification of 4,500 of in the case where the steel plate of FIG. 7 is further heated to 670° C. and sequently cooled therefrom.

FIG. 9 is a photograph showing a structure by the observation with an electron microscope of a magnification of 4,500 in the case where the steel plate of FIG. 8 is subjected to a tempering treatment at 575° C.

FIG. 10 is a photograph showing a structure by the observation with must be electron microscope of a magnification of 4,500 in the case where the steel plate of FIG. 7 to directly 6to 575° C. and sequently cooled therefrom.

FIG. 11 is a photograph showing a structure by the observation with an electron microscope of a magnification of 4,500 in the case where a 3%-Ni series hot-rolled steel plate is subjected to the heat-treatment method of the present invention.

FIG. 12 is a photograph showing a structure by the observation with an electron microscope of a magnification of 4,500 in the case where a 3%-Ni series hot-rolled steel plate is subjected to hardening and tempering treatments.

DESCRIPTION OF THE PREFERRED EMBODIMENT

The essential feature of the present invention resides in producing a structure consisting of a fine ferrite and tempered martensite, wherein a stable ultrafine austenite or cementite is precipitated by subjecting a hot-rolled steel plate containing necessary elements in the ranges as above mentioned or a steel plate obtained by hardening or air-cooling the hot-rolled steel plate to special heating and cooling treatments to and from a temperature between the Ac_1 transformation point and the Ac_3 transformation point and thereupon subjecting the thus treated steel plate to a tempering treatment, which enables the steel to display an excellent toughness at a very low temperature.

It was manifested by the investigations made by the inventors of the present invention that a relatively good toughness could be displayed even at low temperature, when a tough steel containing Ni in a relatively great amount, as a 9%-Ni steel or a 6%-Ni steel, was subjected to hardening and tempering treatments. However, it was further discovered that, if a hot-rolled steel plate having the composition as above mentioned was subjected to a heat treatment specified by the present invention, that is, heating to a temperature within a range between the Ac_1 transformation point and the Ac_3 transformation point and sequent quenching or air-cooling from the said temperature, there could be obtained such an excellent toughness at low temperature and workability as was never seen at any conventional Ni-containing steel as 9%-Ni steel or 6%-Ni steel, whereby the application of steel for use at low temperature was widely spread. Further, as such a remarkable improvement in the toughness at low temperature as above mentioned could be obtained without adding new alloy element, that is, with a steel having the composition in the same or even smaller ranges as the conventional 9%-Ni steel or 6%-Ni steel, a very economical production of an excellent tough steel for use at low temperature was made possible.

In the process of manufacturing the steel of the present invention a melting may be carried out in any well known steelmaking furnace such as converter, open-hearth furnace, electric furnace, high frequency furnace and the like. There is no problem in the point of melting. A molten steel prepared by melting in any of the said furnaces is regulated in its composition by adding alloy elements as required and is made to a hot-rolled steel plate through the steps of ingotting, slabbing and hot-rolling. Then, the thus obtained hot-rolled steel plate is heat-treated as specified by the present invention.

The starting material of the present invention is a hot-rolled steel plate as above mentioned. In the present invention, the hot-rolled steel plate may be immediately subjected to the special heat-treatment of the present invention (a solid solution treatment may be carried out prior to this special heat-treatment) or, as another method, the hot-rolled steel plate may be heated at first to a temperature above the Ac_3 transformation point but below the crystal grain coarsening temperature, with or without the solid solution treatment to be carried out prior to this heat-treatment. In this case, it is not desirable to heat the steel plate to a temperature above the crystal grain coarsening temperature, because the toughness of the steel will be deteriorated thereby. This heating is followed by a cooling with a cooling rate greater than an air-cooling, that is, quenching, or by an air-cooling. The structure of the steel after this treatment becomes a martensite structure or a mixed structure of martensite and bainite (or a mixed structure of martensite, bainite and ferrite according to circumstances).

The steel plate as hot-rolled or further subjected to the heat-treatment to a temperature above the Ac_3 transformation point and sequent quenching or air-cooling as above mentioned is further successively heated to a temperature in the range between the Ac_1 transformation point and the Ac_3 transformation point and sequently quenched or air-cooled.

The essential feature of the present invention is just this special heat-treatment, that is, a heating to a temperature between the Ac_1 transformation point and the Ac_3 transformation point and sequent cooling from the said temperature. By this heat-treatment a fine structure of the steel is produced, whereby the toughness as well as the workability of the steel can largely be improved without reducing the strength thereof at low temperature. That is, when the above-mentioned steel plate as hot-rolled or further subjected to the heat-treatment to a temperature above the Ac_3 transformation point and sequent quenching or air-cooling is further successively heated to a temperature in the range between the Ac_1 transformation point and the Ac_3 transformation point, a fine austenite enriched in C, Ni, Mn and N present as an impurity is precipitated in an insular form at an old martensite crystal grain boundary, old austenite crystal grain boundary or ferrite subgrain boundary with the help of an effect of accelerating the diffusion by the presence of a great number of dislocation groups in the quenched or air-cooled structure resulted from the previous treatment, and this austenite is equilibrated with well annealed and fine ferrite having an excellent toughness, resulting in the formation of a mixed structure of them. In the present invention it is an indispensable condition that the mixed structure of austenite and ferrite is produced by the heating to a temperature between the Ac_1 transformation point and the Ac_3 transformation point. The most preferably range in the above-mentioned heating to a temperature between the Ac_1 transformation point and the Ac_3 transformation point is 620° to 800° C. in the case of the steel having the composition range as above mentioned. Through the sequent quenching or air-cooling a mixed structure of ferrite and fine insular martensite can be obtained. The quenching or air-cooling from the above-mentioned temperature range may be carried out once or may be repeated several times. By repeating the said treatment several times the martensite structure is further refined, whereby the toughness of the refined steel is all the more improved. The medium for use in the quenching may be water, oil, mist or any other which can perform a compulsory cooling. There is no particular limitation in the medium to be selected.

The thus heat-treated steel plate is then tempered at a temperature below the Ac_1 transformation point, preferably in the range of 450° to 600° C. By this tempering treatment austenite is again dispersedly precipitated in a very fine form in the fine martensite islands, and there is finally produced a mixed structure of a pure ferrite resulted from the advanced tempering, a tempered martensite and an extremely fine temper formed austenite, whereby the toughness of steel at lower temperature is largely improved, without causing the reduction of the strength.

However, in the case of the steel containing Ni in a small amount, for instance, less than 4.0 percent and less than 3.5 percent Mn, and the sum of Ni and Mn being less than 4.5 percent, there is found no appearance of the temper formed austenite, but the precipitation of fine cementite in the above-mentioned islands of tempered martensite. The ferrite matrix is also purified, on account of the tempering being well advanced, and becomes a structure rich in the toughness too.

In the following there will be briefly explained the reasons of having limited the amounts of alloy elements to be contained in the steel of the present invention.

C is effective on improving the hardenability and further elevating the stability of austenite at low temperature, as being absorbed into the austenite precipitated during the tempering treatment. However, if the C content is too much, an amount of solid solution carbon in the ferrite matrix will increase, which impairs not only the toughness, but also the weldability of steel. Therefore, the C content was limited to less than 0.2 percent.

Si is an element necessary for steelmaking and is usually contained in an amount of more than 0.05 percent. However, if exceeds 0.4 percent, there appears the tendency of the toughness being deteriorated.

It is well known that Ni is a useful element for improving the toughness and strength of steel. Further, it serves to stabilize the temper formed austenite at low temperature, as being ab-

sorbed into austenite during the tempering treatment. However, if Ni is added too much, it becomes expensive. Therefore, the upper limit thereof was limited to 10.0 percent. On the other hand, if it is less than 1.5 percent, the structure which is the object to be attained by the present invention can not be obtained. Therefore, it is necessary to contain Ni in an amount of at least more than 1.5 percent, and the preferable range is 4.5 to 9 percent.

Mn serves to not only improve the hardenability, but also, like Ni and N, stabilize a very fine austenite precipitated during the tempering treatment and elevate the toughness and strength of the ferrite matrix. However, if it is contained too much, it stabilizes carbide up to a considerably high temperature. Consequently, the upper limit thereof was limited to 5.0 percent. Further, Mn is useful as an element which can replace Ni. Therefore, the range of adding Mn is to be properly decided depending on the Ni content. For instance, if Ni is contained in a range of 4.0 to 7.5 percent, it is preferably to contain Mn in an amount of more than 0.9 percent. But, in case Cu is added, the lower limit of Mn can be extended to 0.5 percent. On the other hand, in case the Ni content is less than 4.0 percent, there is a possibility of austenite being precipitated, if Mn is added in relatively large amount, that is, more than 1.0 percent. But, in this case, that is, in the case of the Ni content being less than 4.0 percent, there is almost precipitated cementite, if the sum of Ni and Mn is less than 4.5 percent. Therefore, in adding Mn, the above-mentioned conditions must be taken into consideration in order to obtain the desired structure. Further, in case Ni is contained in a large amount, Mn may be added in a small amount. At least with 0.1% Mn, the object of the present invention can be achieved. Therefore, the lower limit of Mn was decided to 0.1 percent.

Mo has effects of extending the optimum tempering temperature to the higher side, refining the distributed states of temper formed austenite grains and promoting the diffusion of Mn, C and N. The addition of Mo is also effective to prevent temper embrittlement. For this purpose Mo must be added in a range of 0.05 to 1.0 percent. The same effect can also be achieved, even when a part or the whole of Mo is replaced by W.

Cu may be added, as occasion demands, in order to improve the corrosion resisting property and the toughness of the steel. Like Ni and Mn, also Cu is thought to be effective on stabilizing the temper formed austenite and strengthening some solid solution ferrite matrix itself. For this purpose it may be added in an amount of less than 2 percent.

Cr is added, as occasion demands, in order to improve the strength of steel. Further, it is useful for extending the optimum tempering temperature to the higher side. It is necessary to be added in an amount of 0.1 to 1.5 percent.

Al is necessary to fix nitrogen contained in steel as an impurity, besides being added as a deoxidizer. Al may be replaced by at least one of other nitride-forming elements such as Be, Nb, V and Ta and the like. If the Al content is, however, too much, the impact property at low temperature of steel is deteriorated. Therefore, Al was limited to 0.05 percent in the form of acid-soluble one.

Further, in the present invention at least one element selected from the group consisting of less than 0.20% V, less than 0.2%Nb, less than 0.1% Zr, less than 0.1% Ti and less than 0.005% B for the purpose of particularly imparting the strength to steel and promoting the effect of refining crystal grains.

In the following the examples of the present invention shall be shown.

EXAMPLE 1

In table 1 there are shown mechanical properties of about 6% Ni-containing steels, subjected to the heat treatment of the present invention. All samples consisting of steel plates having a prescribed thickness respectively, as are shown in table 1, have been prepared according to conventional melting process, ingotting, slabbing and hot-rolling.

Among them, the samples Nos. 6A and 6B are steels which contain 6% Ni and 1.7% Mn and are obtained by directly subjecting a hot-rolled steel to the L-treatment (the treatment of heating and cooling to and from a temperature between the Ac_1 transformation point and the Ac_3 transformation point). The L-treatment is followed by the T-treatment (termpering at a temperature below the Ac_1 transformation point). In the case of the sample No. 6A the tempering temperature was 550° C. and in the case of the sample No. 6B 600° C. Both samples show an excellent toughness at low temperature respectively.

The samples Nos. 6C to 6G are those obtained by subjecting a hot-rolled steel plate to the Q-treatment at 800° C. for 1 hour (the treatment of heating to and cooling from a temperature above the Ac_3 transformation point) followed by the L-treatment at 670° C. and thereafter by the T-treatment at 500° to 650° C. respectively. The sample No. H is a reference steel, which was not subjected to the L-treatment. Concerning mechanical properties of each sample of No. 6C to 6G treated according to the method of the present invention, there are shown excellent values for the toughness at low temperature in spite of a variety of tempering temperature. The reference steel No. 6H shows only a value of less than a half of that of the sample No. 6E of the present invention, even though the sample No. 6H was subjected to the T-treatment under the same conditions as the sample No. 6E. From this comparison the above mentioned will be easily understood. Further, by the application of the method of the present invention the range of tempering temperature can be largely widened. In FIG. 1 there are shown the relations between impact value and tempering temperature on steels of the above-mentioned samples Nos. 6C to 6G (shown by a ○ mark) as compared with steels treated according to conventional methods, in which the L-treatment was omitted (shown by a ○ mark). As is evidently seen from this figure, according to the treating method of the present invention a tempering treatment for obtaining high impact values can be carried out extending over a far wider range, as above mentioned, whereby a difficulty in the producing process that the tempering treatment must be carried out under much narrower conditions, as was heretofore, can be eliminated. Moreover, the samples Nos. 6C to 6G show a very low yield ratio respectively, which is particularly manifest in contrast to the sample No. 6H. It can be, therefore, concluded from the foregoing that steels treated by the method of the present invention are superior in the workability to conventional steels.

The samples Nos. 6I to 6N show the results of steels having the Mn content reduced to 1.10 percent being heat-treated, wherein the samples Nos. 6I to 6K are those treated according to the method of the present invention, while the samples Nos. 6L to 6N are those treated according to conventional method. The comparison of both groups shows clearly the quite same tendency with respect to the toughness at low temperature and the workability of steel as is seen at the comparison of the samples Nos. 6C to 6G with No. 6H. The samples Nos. 6O and 6P are steels, which contain Cu, and show that the high toughness at low temperature can be obtained, even though the Mn content is reduced to about 0.5 percent if the steel is treated according to the method of the present invention (for instance, No. 6O). At last, the samples Nos. 6Q and 6R are steels, wherein Nb is added. In this case too, an excellent toughness at low temperature can be imparted to steel, if it is heat-treated according to the method of the present invention (for instance No. 6Q).

As above mentioned, in the steels subjected to the heat treatment of the present invention the toughness at low temperature is remarkably improved and further the workability is elevated, as the yield stress is reduced, that is, the yield ratio is lowered, while there is seen no change in the tensile strength. Usually, in the said steel which contains a relatively large amount of Mn, exceeding 1.0 percent and is further high in the Mo content, cementite remains till to the high temperature side at the time of tempering, whereby the toughness is impaired. In this case, if the tempering is carried out at a high

TABLE 1.—HEAT TREATMENTS AND MECHANICAL PROPERTIES OF 6%-Ni SERIES STEELS

Steel No.	Chemical composition (wt. percent) C	Si	Mn	Ni	Mo	Sol. Al	Other element	Plate thickness, mm.	Heat treatment Q	L	T	Mechanical property σY, kg./mm.²	σB, kg./mm.²	Y.R. (σY/σB), percent	Elongation Total elongation, percent	Uniform elongation, percent	vE-196° C.	Hv
6 A	0.07	0.23	1.70	6.0	0.20	0.15	Balance Fe.	12		700° C.×60 mm. WQ	550 °C.×60 mm. WQ						25.5	
6 B	0.07	0.23	1.70	6.0	0.20	0.15	do	12			600 °C.×60 mm. WQ						26.2	
6 C	0.07	0.23	1.70	6.0	0.20	0.15	do	12	800 °C.×60 mm. AC.	670 °C.×60 mm. WQ	500 °C.×60 mm. WQ	77.9	89.9	0.87	24.2	12.5	5.75	
6 D	0.07	0.23	1.70	6.0	0.20	0.15	do	12	do	do	550 °C.×60 mm. WQ	71.7	83.6	0.86	28.4	15.1	31.3	
6 E	0.07	0.23	1.70	6.0	0.20	0.15	do	12	do	do	600 °C.×60 mm. WQ	69.9	81.4	0.86	29.8	15.3	29.8	
6 F	0.07	0.23	1.70	6.0	0.20	0.15	do	12	do	do	625 °C.×60 mm. WQ	56.4	87.4	0.64	31.9	20.1	26.0	
6 G	0.07	0.23	1.70	6.0	0.20	0.15	do	12	do	do	650 °C.×60 mm. WQ	57.0	98.5	0.58	26.0	15.3	9.0	
6 H	0.07	0.23	1.70	6.0	0.20	0.15	do	12	do	do	600 °C.×60 mm. WQ	69.6	77.8	0.96	24.9	10.8	13.0	
6 I	0.09	0.23	1.10	5.94	0.21	0.01	do	13	do	do	500 °C.×60 mm. WQ	70.7	82.9	0.85	24.2	13.8	24.4	
6 J	0.09	0.23	1.10	5.94	0.21	0.01	do	13	do	do	600 °C.×60 mm. WQ	65.6	75.7	0.86	27.4	16.4	27.1	
6 K	0.09	0.23	1.10	5.94	0.21	0.01	do	13	do	do	625 °C.×60 mm. WQ	55.2	80.2	0.70	29.9	22.3	23.8	
6 L	0.09	0.23	1.10	5.94	0.21	0.01	do	13	do	do	500 °C.×60 mm. WQ	79.5	84.2	0.94	20.5	7.6	6.5	
6 M[1]	0.09	0.23	1.10	5.94	0.21	0.01	do	13	do	do	600 °C.×60 mm. WQ	69.4	76.5	0.91	26.4	14.6	18.8	
6 N[1]	0.09	0.23	1.10	5.94	0.21	0.01	do	13	do	do	625 °C.×60 mm. WQ	63.3	76.1	0.84	26.0	15.6	26.1	
6 O	0.064	0.11	0.52	6.30	0.22	0.01	Cu 0.84; balance Fe.	13	800 °C.×60 mm. WQ	do	600 °C.×60 mm. WQ						22.5	236
6 P[1]	0.064	0.11	0.52	6.30	0.22	0.01	do	13	do	do	do						10.0	250
6 Q	0.10	0.23	1.70	6.0	0.20	0.01	Nb 0.05; balance Fe.	13	800 °C.×60 mm. AC.	640 °C.×20 mm. WQ	do						17.5	297
6 R	0.10	0.23	1.70	6.0	0.20	0.01	do	13	do	do	625 °C.×60 mm. WQ						12.3	278

[1] Steels treated according to conventional methods.

Remarks: Heat treatment: Q means a treatment of heating and cooling to and from a temperature above the Ac_3 transformation point; L means a treatment of heating and cooling to and from a temperature below the Ac_1 transformation point; T means a tempering treatment at a temperature below the Ac_1 transformation point; °C. is temperature, mm. is heating time (minute), WQ is quenching by water, and AC is cooling by air. Mechanical property: σY is Yield stress; σB is Tensile strength; Y.R. (σY/σB) is Yield ratio; $vE_{-196°C.}$ is V-notch Charpy impact test value-196°C.; Hv is Vickers hardness.

temperature in order to prevent the above-mentioned phenomenon, the temper formed austenite becomes unstable, on cooling resulting in a deterioration of the toughness thereby. However, if the steel is treated according to the method of the present invention, austenite is precipitated very fine and dispersedly in the steel and in addition thereto the structure is very stable, whereby an excellent toughness at low temperature can be obtained. Further, as reasons of improving the toughness of steel there can be enumerated the following facts that by the heat treatment of the present invention the tempered structure becomes fine and the ferrite matrix becomes a pure one as a result of the tempering thereof being well progressed.

In the following the structures of steels heat-treated according to the present invention shall be explained in reference to the attached figures (photograph). The samples and the treating methods applied correspond to the above-mentioned samples Nos. 6E and 6H. All figures show microphotographs of 4,500 magnifications respectively. FIG. 2 shows a sample subjected to the following heat treatment: a heating to 800° C. for 1 hour with a sequent air cooling therefrom, wherein it is evidently seen that the structure consists of martensite and partly bainite. FIG. 3 is a photograph showing a structure obtained by further subjecting the steel having the structure as shown in FIG. 2 to the L-treatment of heating to 560° C. for 1 hour and a sequent water cooling. From this photograph it is clear that as a result of the L-treatment there is produced a structure, wherein a martensite transformed from precipitated austenite enriched in C, Ni, Mn and N and precipitated finely in an insular form at an old martensite grain boundary, an old austenite grain boundary or a ferrite subgrain boundary is equilibrated with ferrite, forming a mixed structure of them. The most important feature of the present invention lies just in producing the above-mentioned structure. When the steel having the structure as above mentioned is further subjected to the T-treatment of heating the steel to 600° C. for 1 hour with a sequent water cooling, there is dispersedly precipitated an ultrafine austenite in the tempered martensite, as shown in FIG. 4.

This will be more clearly observed, when compared with the structure of the reference sample shown in FIG. 5. That is, FIG. 5 shows a structure of the steel treated according to a conventional method, wherein the Q treated steel of FIG. 2 was directly subjected to the T-treatment at 600° C., resulting in the production of a coarse precipitated austenite.

EXAMPLE 2

In table 2 there are shown the heat-treating methods of 9% Ni-containing hot-rolled steel plates and mechanical properties obtained thereby, wherein the sample No. 9B is a steel treated according to a conventional method, that is, not subjected to the L-treatment. From the comparison with the sample 9A of the present invention it is clearly seen that the sample No. 9B is far lower in the toughness at low temperature than the sample No. 9A and also in the workability inferior to the latter, as is shown by a higher yield ratio. Further, from FIG. 6 showing the relations between impact value and tempering temperature on the steel shown by the sample No. 9B as compared with the steel treated according to the method of the present invention, it is seen that in the case of the reference steel (shown by a ○ mark) the range of proper T-treatment, that is, the range of tempering temperature capable of obtaining high impact value is very narrow, while in the case of the steel of the present invention the said range is far wider, indicating that an excellent toughness can be obtained in the wide range of tempering temperature.

The structures of steels subjected to various heat-treating methods in the present example are as shown by micro photographs of 4,500 magnifications in FIGS. 7 to 10 respectively. FIG. 7 is a microphotograph showing the structure obtained by the Q-treatment. From this photograph it is seen that the structure of this case consists of a mixed structure of martensite and partly bainite. FIG. 8 shows the structure obtained by subjecting the steel having the structure shown in FIG. 7 to

TABLE 2.—9%-Ni SERIES STEELS

Steel No.	Chemical composition (wt. percent) C	Si	Mn	Ni	Mo	Sol. Al	Other element	Plate thickness, mm.	Heat treatment Q	L	T	Mechanical property σ_y, kg/mm.^{2}	σ_B, kg./mm.^{2}	Y.R. (σ_Y/σ_B),	Elongation Total elongation, percent	Uniform elongation, percent	vE-196° C.
9 A	0.04	0.25	0.43	9.06	.....	0.01	Balance Fe	13	800° C.X60 mm. AC	670° C.X60 mm. WQ	575° C.X60 mm. W	63.2	73.1	0.87	30.0	13.4	20.5
9 B 1	0.04	0.25	0.43	9.06	.....	0.01	Balance Fe	13	800° CX60 mm. PQ	.....	.....	70.0	75.3	0.93	24.4	10.5	7.9

the L-treatment, wherein it is clearly seen that fine martensite in the insular form is precipitated in the ferrite matrix. When further subjecting the sample of FIG. **8** to the T-treatment, there can be obtained a structure shown by the photograph in FIG. **9**, wherein the tempered martensite is formed in the ferrite matrix, where the tempering made progress, in said tempered martensite ultra fine austenite being precipitated. For the purpose of comparison there is shown in FIG. **10** the final structure of the sample No. 9B, which was not subjected to the L-treatment. From this figure it is quite evident that the temper formed austenite has a coarse structure, as compared with the structure shown in FIG. **9**.

EXAMPLE 3

In table 3 there are shown mechanical properties, particularly the toughness and strength, of about 3.0% Ni-containing hot-rolled steel plates manufactured by a conventional method, as subjected to the Q-treatment and the T-treatment with or without the L-treatment.

The samples Nos. 3A and 3C and Nos. 3F and 3G are the steels treated according to the method of the present invention, including the L-treatment, while the samples Nos. 3D, 3E and 3H are those treated to a conventional method, wherein the L-treatment is not included. From the comparison of both groups it is clear that the steels of the present invention are far superior in the toughness to conventional steels. That is, in the steels treated according to the method of the present invention there is produced a very fine tempered structure, wherein cementite is finely precipitated in islands of the tempered martensite. Further, as the tempering of the ferrite matrix is made progress and there are produced pure ferrite of fine grains which does not contain cementite, a structure having an excellent toughness can be obtained. FIG. **11** is a microphotograph of 4,500 magnifications of the final structure of the sample No. 3A, from which the above-mentioned fact is manifested to be true. FIG. **12** is also a microphotograph of the final structure of the sample No. 3D which was not treated according to the method of the present invention, showing that a coarse cementite is being extensively precipitated, which established that the steel treated according to the method of the present invention is superior to the conventional steel.

As is above explained, the steel heat-treated according to the method of the present invention has an excellent toughness at low temperature without being attended with a reduction of the strength, can be easily produced, because the optimum tempering temperature extends over a wide range and further can be used for various purposes, as the workability at normal temperature is strikingly improved. However, the present invention is not limited to the examples above-mentioned, but may be modified variously within the scope of the objects of the present invention.

What is claimed is:

1. A heat-treating method for producing a steel having an excellent toughness at an extremely low temperature and a high strength and good workability from a steel consisting essentially of less than 0.20 wt. % C. 0.05 to 0.40 wt.% Si, 0.10 to 5.0 wt.% Mn, 1.50 to 10.0 wt.% Ni as basic components comprising subjecting a hot-rolled steel plate having the said composition to a heat-treatment comprising heating to a temperature between the Ac_1 transformation point and the Ac_3 transformation point and a subsequent cooling from the said temperature at least at an air cooling rate and thereupon tempering the steel plate at a temperature below the Ac_1 transformation point.

2. A method according to claim **1**, wherein the heat-treatment comprising the heating of the hot-rolled steel plate to a temperature between the Ac_1 transformation point and the Ac_3 transformation point and the subsequent cooling from the said temperature is carried out repeatedly more than one time.

3. A method according to claim **2**, wherein prior to said heat-treatment, said hot-rolled steel plate is subjected to a solid solution treatment.

4. A heat-treating method for producing a steel having an excellent toughness at an extremely low temperature and a

TABLE 3.—3%-Ni SERIES STEELS

Steel No.	Chemical composition (wt. percent) C	Si	Mn	Ni	Mo	Sol. Al	Other element	Plate thickness, mm.	Heat treatment Q	L	T	Mechanical property vTrs (° C.)	Hv
3 A	0.10	0.24	0.69	3.03		0.36	Balance Fe.	11	900 ° C.×60 mm. WQ	720 ° C.×60 mm. WQ	500 ° C.×60 mm. WQ	−190	205
3 B	0.10	0.24	0.69	3.03		0.36	do	11	do	do	550° C.×60 mm. WQ	−175	195
3 C	0.10	0.24	0.69	3.03		0.36	do	11	do	do	600° C.×60 mm. WQ	−165	181
3 D [1]	0.10	0.24	0.69	3.03		0.36	do	11	960° C.×60 mm. WQ		500° C.×60 mm. WQ	−160	249
3 E [1]	0.10	0.24	0.69	3.03		0.36	do	11	do		600° C.×60 mm. WQ	−150	207
3 F	0.045	0.21	4.45	2.95	0.21	0.015	do	11	800° C.×60 mm. WQ	670° C.×60 mm. WQ	do	[2] 11.0	253
3 G	0.045	0.21	4.45	2.95	0.21	0.015	do	11	do	700° C.×60 mm. WQ	do	[2] 15.6	260
3 H [1]	0.045	0.21	4.45	2.95	0.21	0.015	do	11	do		do	[2] 8.1	235

[1] See Table 1.
[2] $vE_{-196°}$ c. (kg. m./cm.).
Remarks: vTrs (° C.) is V-notch Charpy 50% ductile transition temperature. Other signs are of the same meaning as Fig. 1.

high strength and good workability from a steel consisting essentially of less than 0.20 wt.% C. 0.05 to 0.40 wt.% Si. 0.10 to 5.0 wt.% Mn, to 1.50 to 10.0 wt.% Ni as basic components, comprising heating a hot-rolled steel plate having the said composition to a temperature below the austenite grain coarsening temperature above the Ac_3 transformation point and subsequently cooling the steel plate from the said temperature at least at least at an air-cooling rate, then subjecting the steel plate to a heat-treatment comprising heating the steel plate to a temperature between the Ac_1 transformation point and the Ac_3 transformation point and subsequently cooling said steel plate from the said temperature at least at an air cooling rate and thereupon tempering the steel plate at a temperature below.

5. A heat-treating method according to claim 4, wherein the heat-treatment comprising the heating of the steel plate to a temperature between the Ac_1 transformation point and the Ac_3 transformation point and the subsequent cooling from the said temperature at least at an air cooling rate is carried out repeatedly more than one time.

6. A heat-treating method according to claim 4 wherein said hot-rolled steel plate is subjected to a solid solution treatment prior to heating the steel plate to a temperature below the austenite grain coarsening temperature above the Ac_3 transformation point and subsequently cooling the steel plate from the said temperature.

7. A method as claimed in 1 wherein said steel further comprises at least one element selected from the group consisting of 0.05 to 1.0 wt.% Mo, 0.1 to 2.0 wt.% Cu, 0.1 to 1.50 wt.% Cr. less than 1.0 wt.% Nb, less than 1.0 wt.% V and less than 0.05 wt.% sol. Ao, and the rest being Fe and unavoidable impurities.

8. A method as claimed in claim 4, wherein said steel further comprises at least one element selected from the group consisting of 0.05 to 1.0 wt.% Mo. 0.1 to 2.0 wt.% Cu, 0.1 to 1.50 wt.% Cr, less than 1.0 wt.% Nb, less than 1.0 wt.% V and less than 0.05 wt.% sol. Al and the rest being Fe and unavoidable impurities.

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,619,302 Dated November 9, 1971

Inventor(s) KOICHI AOKI ET AL

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

LIST OF CORRECTIONS

(Since the line numbers in the printed patent are not always correctly aligned, the following line indications refer for clarity's sake to the actual line numbers as counted from the top of the respective column)

Column 1 (The line "METHOD OF HEAT-TREATING LOW TEMPERATURE" is considered as line 1)

1. line 9, after "temperature" insert --,--;

2. line 15, after "after" insert --having been--;

3. line 34, change "not still" to read --still not--;

4. line 39, change "used to be detested" to --not favored--;

5. line 71, change "demand" to --demands--.

Column 2

6. line 1, after "impurities" insert --,--;

7. line 19, (first line after "BRIEF DESCRIPTION OF THE DRAWINGS") change "between a temper" to --between temper--;

ORM PO-1050 (10-69)

USCOMM-DC 60376-P69

* U.S. GOVERNMENT PRINTING OFFICE : 1969 O—366-334.

UNITED STATES PATENT OFFICE

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Patent No. 3,619,302 Dated November 9, 1971

Inventor(s) KOICHI AOKI ET AL

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

(continued)

Column 2

8. line 20, change "a toughness" to --toughness--;

9. line 30, rewrite the line "of in the case where the steel of FIG. 2 is further heated and" to read --of the steel of FIG. 2 after having been further heated and--;

10. line 34, rewrite to read --of the steel plate of FIG. 3 after having been sub- --;

11. line 38, rewrite to read --of the steel plate of FIG. 2 which is directly sub- --;

12. line 40, change "a temper-" to --temper- --;

13. line 41, change "a toughness" to --toughness--;

14. line 51, rewrite to read --of the steel plate of FIG. 7 after having been further heated;

15. line 55, rewrite the line to read --of the steel plate of FIG. 8 after having been subjected to a--;

16. line 58, change "must be" to --an--;

17. line 59, rewrite to read --4,500 of the steel plate of FIG. 7 is directly heated to--.

FORM PO-1050 (10-69)

USCOMM-DC 60376-P69

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Patent No. 3,619,302 Dated November 9, 1971

Inventor(s) KOICHI AOKI ET AL

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

(continued)

Column 7

18. In the definition of the symbols appearing below Table 1, lines 1 and 2, replace " L means a treatment of heating and cooling to and from a temperature below the Ac_1 transformation point" by --L means a treatment of heating and cooling to and from a temperature between the Ac_1 transformation point and the Ac_3 transformation point--.

Column 11

19. Claim 4, line 3 of the claim as appearing in the column, change "Mn, to 1.50" to --Mn, 1.50--;

20. Claim 4, last line, after "below" insert --the Ac_1 transformation point--.

Column 12

21. Claim 7, line 5, change "Ao" to read --A1--.

Signed and sealed this 16th day of July 1974.

(SEAL)
Attest:

McCOY M. GIBSON, JR.
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents