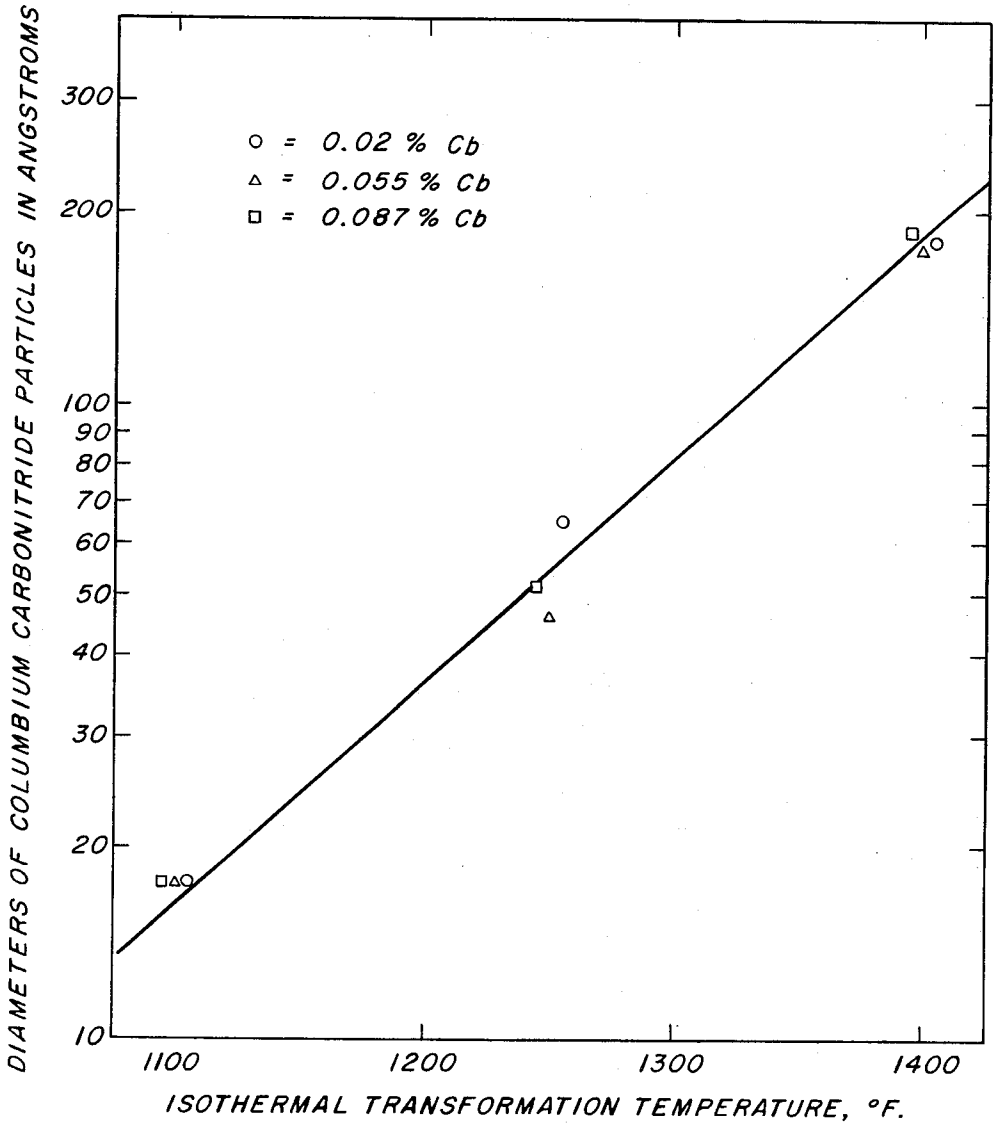
METHOD FOR PRODUCTION OF LOW CARBON STEEL WITH HIGH DRAWABILITY AND RETARDED AGING CHARACTERISTICS

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METHOD FOR PRODUCTION OF LOW CARBON STEEL WITH HIGH DRAWABILITY AND RETARDED AGING CHARACTERISTICS

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ABSTRACT OF THE DISCLOSURE

A sheet steel with exceptionally high drawability and retarded aging characteristics is produced by adjusting the melt to less than 0.50% Mn, 0.002 to 0.06% C, less than 150 p.p.m. oxygen and adding Cb to combine with the C and N present in the melt. The slab cast from the melt is then hot rolled above the A_3 temperature and heat-treated to produce a critical coarse dispersion of carbonitrides. The resultant sheet is then cold reduced from 50 to 90% and soaked at temperatures within the range 1200° F. to the ferrite to austenite transformation temperature of the steel to develop desired crystallographic textures.

This invention relates to a method of producing low-carbon steel sheets having improved deep drawing and non-strain-aging characteristics by careful control of both compositional and processing variables.

At present, two basic types of low-carbon sheet steels are used commercially for press forming. These are low-carbon rimmed steels and aluminum-killed steels. The low-carbon rimmed steels are more economical for both the producer and the user and have better surface characteristics than the aluminum-killed steels; however, they do not have the good deep-drawing properties of aluminum-killed steels and they are susceptible to strain-aging. Strain-aging of rimmed steel sheets is responsible for the formation of unsightly "Lüders-lines" or "stretcher-strain markings" on the surface of press-formed parts.

A sheet steel exhibiting excellent press formability and low susceptibility to strain aging has recently been disclosed in U.S. Pat. 3,522,110. These properties are achieved, in the main, by limiting the C content to a value less than 0.02% and the oxygen content to less than 0.015% and employing Ti in an amount equal to at least four times the C content. Goodman and Hu U.S. Pat. 3,709,744 have shown that if certain critical processing parameters are employed, and Mn is limited to a value of 0.15% max., then similar high drawability may be achieved without the use of Ti and with significantly higher C contents (and therefore higher yield strengths). However, a serious disadvantage of this latter low Mn steel is that, unlike the Ti-stabilized steel of Shimizu et al. (U.S. Pat. 3,522,110) and the commercial aluminum killed steels, it exhibits a propensity for strain aging. A disadvantage which could, as is well known in the art, be overcome by the addition of nitride forming elements as Al, Ti, Cb, etc.

The press formability of steel sheets is dependent on two properties: the drawability (the ability to draw-in steel from the flange area of a pressed part into the recessed die-cavity of the die) and the stretchability (the ability of the steel to stretch, under biaxial tension, to the contours of a punch and/or die cavity). Drawability is related to the plastic anisotropy of steel sheets. A measure of the plastic anisotropy is the plastic-strain ratio, r, which can be determined from sheet tension tests, and

which is defined as the ratio of the true width strain (ϵ_w), to the true thickness strain (ϵ_t), i.e.,

$$r=\frac{\epsilon_w}{\epsilon_t}$$

$$r=\frac{\ln (W_o/W_f)}{\ln (L_f W_f/L_o W_o)} \quad (1)$$

where W and L are the specimen width and length, respectively, and the subscripts o and f refer to the original and final measurements, that is, before and after plastic straining, respectively. (The extent of plastic strain in the length direction is usually 15 to 20%.) For most commercial low-carbon steel sheets, the plastic-strain ratio varies with test direction in the plane of the sheet. Therefore, the plastic-strain ratio is usually measured at 0, 45, and 90 degrees to the sheet rolling direction. The normal plastic anisotropy of the sheet is then described by the average plastic-strain ratio, $\bar{r}$, which is defined as follows:

$$\bar{r}=(r_0+2r_{45}+r_{90})/4 \quad (2)$$

The magnitude of $\bar{r}$ has been shown to correlate with drawability, and therefore $\bar{r}$ is often used to predict the depth of draw which can be achieved during drawing operations.

The variation of r in the plane of the sheet is termed the planar anisotropy. The parameter Δr has been shown to correlate with the tendency of a sheet to form ears during deep drawing. Δr is defined as:

$$\Delta r=(r_0-2r_{45}+r_{90})/2$$

Not only does the magnitude of Δr indicate the severity of earing but the sign of Δr predicts the position of ears in the drawn part, with respect to the original sheet rolling direction. Thus, positive values of Δr are associated with ears at 0 and 90 degrees to the rolling direction while negative values of Δr are related to 45-degree earing.

Recently, the deep-drawability of steel sheets has been related to the type and intensity of crystallographic textures, as determined by X-ray techniques. Good deep drawability has been associated with a large number of grains of the steel sheet oriented such that (111) and (112) planes are parallel to the sheet surface. Conversely, poor deep drawability has been associated with a large number of grains oriented with (100) and (110) planes parallel to the sheet surface. Hence, the intensity of these texture components [often expressed as a ratio, i.e. $I_{(111)}/I_{(100)}$ or $I_{(111)}+I_{(112)}/I_{(100)}+I_{(110)}+I_{(310)}$] has been used to assess plastic anisotropy and hence predict deep drawability.

The stretchability of steel sheets is determined largely by the structure of the sheet, i.e. the grain size, the size and form of non-metallic inclusions and the concentration of alloying and residual elements. High values of the plastic strain-hardening exponent, n, are favorable for high stretchability. The strain-hardening exponent, n, is determined experimentally as the slope of the log true stress versus log true strain relationship in the region of uniform strain. For commercial low-carbon sheet steels, n usually varies from 0.200 to about 0.270 in drawing-quality steels. Low values of the lower yield stress are usually required for press-forming sheet steels, because the strain-hardening exponent usually increase as the low yield stress decreases.

It is, therefore, an object of this invention to produce a steel sheet having a crystallographic texture with a predominance of (111) and (112) planes in the plane of the sheet.

It is another object to provide a steel composition which, when processed by the method of this invention, will yield extremely high $\bar{r}$ values, i.e., values greater than about

1.50, and preferably greater than 1.70, together with satisfactory n values.

A further object of this invention is to provide a steel composition which exhibits a combination of high $\bar{r}$ values

Another objective of this invention is to provide a steel composition and process for producing sheets which minimixes the value of Δr and consequently minimizes the extent of earing after pressing into a commercial part.

This invention is based on the discovery that a number of compositional and processing variables are critical for providing maximum $\bar{r}$ values, minimum Δr values, freedom from strain-aging and satisfactory values of n and lower yield strength. The manganese and oxgen contents of a steel melt are adjusted to below 0.50% Mn and 0.015% oxygen. Further enhancement of the $\bar{r}$ values may be achieved if the Mn content is adjusted to below 0.15% in accordance with the teachings of Goodman and Hu, U.S. Pat. 3,709,744, the disclosure of which is incorporated herein by reference. Cb is then added to the melt in sufficient quantity (discussed more fully below) depending on the level of C and N present in the steel. It is desirable that the Cb be added after the oxygen has been lowered to the specified level so that the combination of Cb with oxygen will be minimized. In order to prevent the yield strength from becoming unduly high due to the presence of Cb, it is necessary to limit the C content to a value somewhat lower than that of Ser. No. 15,018, i.e. to between 0.002 and 0.06%. The cast slab should then be hot rolled at a temperature sufficiently high to ensure that the final hot-rolling pass be made above the temperature at which proeutectoid ferrite will form. It has now been found, that in order to achieve the beneficial effects of the Cb addition, it is essential that the hot rolled sheet be heat treated to cause the dispersed carbonitrides to coalesce, so that substantially all (preferably greater than 95%) the particles fall within the range 40 A. to 500 A., with a predominant portion being greater than 125 A. in size. After cooling to room temperature, the sheet must be cold reduced between 50 and 90%, and soaked at temperatures above 1200° F. but below the temperature at which the steel transforms to austenite, for at least 12 hours. The compositional limits of the steels of this invention are summarized in Table I, below:

TABLE I

Element	Composition, wt. percent	
	Broad	Preferred
Carbon	0.002 to 0.06	0.005 to 0.03.
Manganese	0-005 to 0.50	0.005 to 0.15.
Silicon	0.06 maximum	0.06 maximum.
Sulfur	0.002 to 0.03	0.002 to 0.015.
Phosphorus	0.02 maximum	0.01 maximum.
Oxygen	0.015 maximum	Do.
Nitrogen	0.001 to 0.01	0.001 to 0.005.
Columbium	0.06 to 0.60	0.06 to 0.20.
Iron	Balance except for incidental steelmaking impurities	

The criticality of these compositional and processing parameters will be better understood by reference to the following description, the appended claims and the figures, in which

FIG. 1 is a graph depicting the dependence of carbonitride particles size on the temperature of isothermal transformation.

In experiments leading to the instant invention, four steels (Table II) containing varying amounts of columbium were vaccum melted, cast into 100-pound ingots and hot rolled to 1-inch-thick plates. The plates were surface ground to 0.8-inch thickness, soaked at 2300° F. for 1 hour and hot rolled in five passes to 0.100 inch-thick sheet, with the last pass occurring at 1700° F. The sheet was then water spray cooled to 1100° F., cold-rolled 70%, box annealed at 1300° F. for 16 hours and temper rolled 2%. As may be seen in Table III, the columbium containing steels evidenced relatively poor $\bar{r}$ values when compared with the steel of Ser. No. 15,018 (Steel A), the $\bar{r}$ values decreasing with increasing Cb content. Although steels B, C and D contained sufficient Cb to combine with the N present (e.g. U.S. Pat. 2,999,749) they nevertheless exhibited strain aging index values in excess of zero. Electron micrographs of these steels showed that the carbonitrides present were substantially all within the size range of 10 to 160 A, with a predominant portion being less than 100 A. The size distribution was found to be independent of columbium content.

To demonstrate the importance of the temperature of transformation (from austenite to ferrite and carbonitrides) following hot-rolling, samples of steels B, C, and D were isothermally transformed at different temperatures in the following way. Samples of hot-rolled sheets of B, C, and D (hot roll finished at 1700° F. and spray cooled to 1100° F.) were reheated to 2300° F. for 15 minutes and quenched into furnaces of molten salt at 1400°, 1250°, or 1100° F., held at temperature for four hours, removed, and air cooled to room temperature. The steels quenched to 1400° F. and held for four hours were furnace cooled in the salt pot to 1300° F. and then air cooled to ensure complete transformation to ferrite and carbonitrides at temperatures above 1300° F. To eliminate the possibility of variations in the soluble C and N contents between the different samples, the sheets were given an equilibrating anneal by reheating to 575° F. in a muffle furname and holding for 16 hours. The sheets were then shot blasted to remove scale, cold reduced 70% by rolling, and box annealed at 1300° F. for 16 hours. The resulting mechanical properties are listed in Table IV. The coarse austenite grain size resulting from reaustenization, and the rapid transformation from austenite to ferrite due to the quenching yielded a structure substantially different from normal hot rolling. It would therefore be expected that such a procedure would have a deleterious effect on r values of subsequently cold rolled and annealed sheets. However, although these values were relatively low, the consistent improvement with increasing temperature is clearly evident. Similarly, when the transformation temperature was increased a decided improvement in the intensity of the favorable crystallographic texture components [i.e. (111) and (112)] of cold rolled and annealed sheets was noted. Electron micrographs showed that these changes in crystallographic texture and r values were a result of the difference in the size and spacing of the carbonitrides formed at the different transformation temperatures. FIG. 1 shows that the diameter of the carbonitrides, as measured from the electron micrographs, varied markedly with the temperature at which the particles were formed, i.e. the temperature of transformation.

Utilizing these findings, samples of steels B, C and D were hot rolled with a finishing temperature of 1600° F., air cooled on the run-out table of the hot mill to a simulated coiling temperature of 1450° F. (taking about 10 seconds), and cooled at a rate of 50° F. per hour (equivalent to the cooling rate of commercial-sized coils following coiling) to 1100° F., and furnace cooled to room temperature (taking about four hours), the size of the columbium carbonitrides was found to vary from 40 to 500 angstroms. This wide range of carbonitride particle size reflects the wide temperature range over which the particles formed during continuous cooling and is in contrast to the narrow size range for particles formed during an isothermal transformation (FIG. 1). After a cold rolling reduction of 70 percent and a box anneal at 1300° F. for 16 hours, the r values for all three steels were markedly improved (Table V), with the degree of improvement increasing with the concentration of Cb. Also noteworthy is the desirable low Δr values, and the decrease in strain aging index achieved; with steels B and C exhibiting retarded aging characteristics while steel D was essentially nonaging.

TABLE II.—CHEMICAL COMPOSITION OF 1-INCH-THICK HOT ROLLED PLATES

Steel	Composition, percent								
	C	Mn	P	S,	Si	N	Total	O	Cb
A	0.028	0.01	0.002	0.002	0.022	0.005	0.007	0.0049	0.005
B	0.029	0.01	0.002	0.002	0.028	0.006	0.005	0.0054	0.020
C	0.026	0.01	0.002	0.002	0.025	0.008	0.005	0.0052	0.055
D	0.025	0.01	0.002	0.002	0.013	0.009	0.002	0.0130	0.087

aging, low-carbon steel sheet having desirably high r values (>1.50) and low Δr values, and having satisfactory $\bar{n}$ values can be produced by following the prescribed composition and processing limits of this invention. The exceptional qualities of this steel are directly related to the formation of coarse columbium carbonitrides in the hot-rolled sheet which allows the development of excellent crystallographic textures after cold rolling and annealing, and which also inhibits strain-aging.

TABLE III

Steel	Cb, percent	r_0	r_{45}	r_{90}	$\bar{r}$	Δr	n_0	n_{45}	n_{90}	$\bar{n}$	Lower yield stress, K s.i.	Strain aging index, percent
A	<0.005	1.36	1.62	2.10	1.68	+0.12	0.189	0.176	0.176	0.179	44.67	16.0
B	0.02	1.53	1.27	1.86	1.49	+0.43	0.190	0.178	0.174	0.180	48.73	10.9
C	0.055	1.54	1.32	1.83	1.52	+0.36	0.150	0.126	0.117	0.130	51.60	9.4
D	0.087	1.19	1.20	1.47	1.27	+0.14	0.153	0.133	0.117	0.134	53.22	8.1

TABLE IV

Steel	Cb, percent	Transformation temperature, °F.	r_0	r_{45}	r_{90}	$\bar{r}$	Δr	n_0	n_{45}	n_{90}	$\bar{n}$	Lower yield stress, K si.
B	0.02	1,100	1.11	0.72	1.31	0.97	+0.50	0.204	0.186	0.206	0.195	40.55
C	0.055	1,100	0.88	0.65	0.96	0.79	+0.28	0.202	0.172	0.165	0.178	25.30
D	0.087	1,100	0.76	0.62	0.67	0.67	+0.10	0.162	0.110	0.129	0.128	26.70
B	0.02	1,250	1.48	0.68	1.19	1.01	+0.65	0.198	0.185	0.192	0.190	38.60
C	0.055	1,250	0.96	0.71	1.09	0.87	+0.32	0.225	0.207	0.177	0.204	20.96
D	0.087	1,250	0.67	0.79	0.60	0.71	+0.16	0.110	0.120	0.104	0.114	28.80
B	0.02	1,400	1.65	1.17	1.55	1.39	+0.43	0.231	0.216	0.205	0.217	31.40
C	0.055	1,400	1.33	0.99	0.93	1.06	+0.15	0.186	0.163	0.119	0.158	3.60
D	0.087	1,400	0.87	0.95	0.52	0.82	−0.26	0.159	0.123	0.111	0.129	24.70

TABLE V

Mechanical properties of columbium steels hot-roll finished at 1,600° F., and slow cooled from 1,450 to 1,100° F., air cooled to room temperature, cold rolled 70 percent and box annealed at 1,300° F. (16 hrs.)

Steel	Cb, percent	r_0	r_{45}	r_{90}	$\bar{r}$	Δr	n_0	n_{45}	n_{90}	$\bar{n}$	Lower yield stress, K s.i.	Strain aging index, percent	Grain size of annealed sheet, ASTM No.
B	0.02	1.71	1.49	2.24	1.73	+0.48	0.237	0.231	0.231	0.233	40.50	5.4	9.7
CC	0.055	1.58	1.87	2.46	1.94	+0.15	0.231	0.218	0.214	0.220	37.91	4.0	10.0
DD	0.087	1.64	2.15	2.41	2.09	−0.13	0.227	0.216	0.200	0.215	43.15	0	9.9

Although the stoichiometric weight ration of Cb:N in columbium nitride is 6.7:1, a comparison of the strain-aging indices of steels C and D indicates that a greater amount of columbium is required to prevent strain aging due to nitrogen. Thus, since some of the Cb will combine only with the C and will be unavailable to the N, it is necessary to employ Cb in excess of the stoichiometric ratio to produce a steel which is nonaging. Therefore, in order to produce a sheet steel which is essentially non-aging it is preferred that the ratio of Cb:C+N be at least 8:1, wherein all the C and N can be expected to form carbonitrides with the Cb. As would be expected from the inhibiting effect of Cb on grain growth, the annealed steel sheets had fine grain sizes which probably accounted for the low, but satisfactory, n values and the high yield yield strengths achieved (Table V).

However, any problems resulting therefrom, could easily be overcome by judicious control of the Cb, control of the C and N contents and/or by the use of long-time, high temperature anneals to provide grains within the range ASTM 7–8. Such larger grain sizes would provide an increase in n values and a decrease in yield strength without any loss in plastic anisotropy. Therefore, if the compositions are controlled so that the ratio of Cb:C+N is greater than the stoichiometric ratio in columbium carbonitride, all of the carbon and nitrogen can be expected to form carbonitrides. Consequently, due to absence of a yield-point elongation, the temper rolling of low carbon steel sheets, which is currently employed to prevent formation of Lüders lines prior to press forming, could be eliminated.

From the above data, it is clear that a non-strain-

The product of this invention may therefore be produced in the following manner. A steel is melted to the prescribed composition. The oxygen content of the steel is reduced to below about 150 p.p.m. (such as by vacuum-carbon deoxidation and/or by addition of sufficient Al) after which columbium is added to the melt so that the resulting steel ingots or slabs are within the prescribed compositional range. The slabs are then heated for hot rolling to sheet at a sufficiently high temperature (e.g. about 2300° F.) to ensure that the final hot rolling pass is made at a temperature above that at which proeutectoid ferrite will form. The use of columbium offers a further advantage in this regard, over a composition free of such an element, since it will, in general, lower the transformation temperature of the steel. This is desirable in a practical sense, since a lower hot-roll finishing temperature is easier to achieve than the narrower finishing range of the low manganese steels of Ser. No. 15,018, now U.S. Pat. No. 3,709,744. The steel is then heat-treated to cause the formation of coarse carbonitride particles, i.e. with diameters in the range of 40 to 500 A. Thus, for example, this heat treatment can be effected by coiling within the preferred temperature range of 1300° to 1450° F. and cooling slowly (e.g. at rates less than 100° F. per hour, as occurs for tightly wound commercial coils) in air to room temperature. Alternatively, low-temperature coiling (less than 1300° F.) may be employed, followed by annealing at temperatures within the range 1300° to 1850° F. for a period of ¼ to 5 hours, wherein the times employed are generally inversely proportional to temperature. This latter anneal may also be employed in supplement to the former high-temperature coiling procedure.

Hot mill scale is then removed, and the hot-rolled sheet is then reduced 50 to 90% (preferably 60 to 80%) by cold rolling to final gage. The cold-rolled sheet is then either (a) wound into a tight coil and box annealed by slow heating to a temperature which will assure recrystallization (e.g. above 1200° F.) but less than the ferrite to austenite transformation temperature of the steel and soaked for at least 12 hours (preferably more than 16 hours) or (b) the sheet is open wound and annealed at about the same temperatures and for about the same times.

We claim:

1. A method for the production of low carbon, sheet steel exhibiting a combination of exceptionally high drawability and retarded aging characteristics, which comprises:

(a) adjusting the composition of a steel melt so that it consists essentially of, in weight percent

N	0.001 to 0.01
C	0.002 to 0.06
Mn	0.005 to 0.50
Oxygen, max.	0.015
Cb	0.06 to 0.60
Fe	Balance

the C and N being substantially combined with said Cb in a random dispersion as columbium carbonitride,

(b) hot rolling a slab produced from said melt at a temperature sufficiently high to prevent the formation of proeutectoid ferrite during hot rolling,

(c) coiling the resultant hot rolled sheet at a temperature within the range 1300° F. to the A_3 temperature of said steel, followed by slow cooling, to cause the dispersed columbium carbonitrides to coalesce so that the particle size diameters thereof, fall substantially within the range 40 to 500 A., with a predominant portion being greater than 125 A. in size,

(d) cold reducing the heat-treated, hot-rolled sheet to from 50 to 90%, and

(e) soaking the cold reduced sheet at a temperature within the range 1200° F. to the ferrite to austenite transformation temperature of the steel for a period of at least about 12 hours.

2. The method of claim **1**, wherein said slow cooling is conducted at a rate less than 100° F. per hour.

3. The method of claim **2**, wherein said steel is made essentially non-strain aging by employing said Cb in an amount greater than the stoichiometric ratio of Cb:C+N in columbium carbonitride.

4. The method of claim **3**, wherein the ratio Cb:C+N is equal to or greater than 8:1.

5. The method of claim **4**, wherein the drawability of said sheet steel may be materially enhanced by adjusting the Mn content of said steel melt to be less than 0.15%.

6. The method of claim **5**, wherein the N, C and oxygen in step (a) are adjusted to consist essentially of

N	0.001 to 0.005
C	0.005 to 0.030
Oxygen, max.	0.01

7. The method of claim **6**, wherein the contents of Si, S and P are maintained within the following ranges:

Si, max.	0.06
S	0.002 to 0.015
P, max.	0.01

8. A method for the production of low carbon, sheet steel exhibiting a combination of exceptionally high drawability and retarded aging characteristics, which comprises,

(a) adjusting the composition of a steel melt so that it consists essentially of, in weight percent

N	0.001 to 0.01
C	0.002 to 0.06
Mn	0.005 to 0.50
Oxygen, max.	0.015
Cb	0.06 to 0.60
Fe	balance

the C and N being substantially combined with said Cb in a random dispersion as columbium carbonitride,

(b) hot rolling a slab produced from said melt at a temperature sufficiently high to prevent the formation of proeutectoid ferrite during hot rolling,

(c) coiling the resultant hot rolled sheet at a temperature below 1300° F., followed by an anneal at temperatures within the range 1300° F. to 1850° F. for a time of about ¼ to 5 hours, wherein the time employed is, in general, inversely proportional to the temperature of said anneal, to cause the dispersed columbium carbonitrides to coalesce so that the particle size diameters thereof fall substantially within the range 40 to 500 A., with a predominant portion being greater than 125 A. in size,

(d) cold reducing the heat-treated, hot-rolled sheet to from 50 to 90%, and

(e) soaking the cold reduced sheet at a temperature within the range 1200° F. to the ferrite to austenite transformation temperature of the steel for a period of at least about 12 hours.

9. The method of claim **8**, wherein said steel is made essentially non-strain aging by employing said Cb in an amount greater than the stoichiometric ratio of Cb:C+N in columbium carbonitride.

10. The method of claim **9**, wherein the ratio Cb:C+N is equal to or greater than 8:1.

11. The method of claim **10**, wherein the drawability of said sheet steel may be materially enhanced by adjusting the Mn content of said steel melt to be less than 0.15%.

12. The method of claim **11**, wherein the N, C and oxygen in step (a) are adjusted to consist essentially of

N	0.001 to 0.005
C	0.005 to 0.030
Oxygen, max.	0.01

13. The method of claim **12**, wherein the contents of Si, S and P are maintained within the following ranges:

Si, max.	0.06
S	0.002 to 0.015
P, max.	0.01

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