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METHOD FOR PRODUCING DEEP-DRAWING
LOW-CARBON STEEL SHEET

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This invention relates to low-carbon steels having improved drawability. More particularly, the invention is directed to a method of producing low-carbon sheet steel with improved deep-drawing characteristics and high yield strength.

It is well known that aluminum killed steels have excellent drawability. Aluminum killed steels, referred to as "SK" grade steel, are characterized by flattened or pancake-shaped ferrite grains which are crystallographically oriented to provide good drawability. Such grains 20 plane. Therefore, the ideal texture for optimum draware developed in the final sheet product by a properly controlled box annealing process during which selective growth of the favorably oriented grains is effected by the aluminum nitride precipitate. Although the exact mechanism of the process is not fully known, the phenomenon that a critically dispersed second phase can markedly affect the recrystallization and grain growth is frequently observed, and in some cases, successfully employed in metallurgical applications.

However, aluminum-killed steels are relatively expen- 30 sive due not only to the cost of the alloy, but also to the low yield from ingots and high conditioning costs. It is, therefore, desirable to develop a new method for producing deep-drawing sheets of relatively less expensive low-carbon steels.

The drawability of sheet material can be evaluated by simple tension tests. When a strip specimen is pulled to a greater length, its width and thickness are decreased. The plastic strain ratio can serve as an indication of the degree of mechanical anisotropy of the material. This ratio is referred to as the "R value" and is defined as the ratio of percent change in width (ew, the width strain) to the percent change in thickness (et, the thickness strain), i.e.

ness strain), i.e.
$$R=e_{\rm w}/e_{\rm t}$$
 or
$$R=\frac{\ln{(W_{\rm i}/W_{\rm f})}}{\ln{(L_{\rm f}W_{\rm f}/L_{\rm i}W_{\rm i})}}$$

where W and L are the width and length, respectively, of the gauge section, and the subscripts i and f refer to the initial and final measurements (before and after straining) of these dimensions. This expression is based on the assumption that the volume of the gauge section remains constant during testing and it eliminates the direct measurement of the thickness which owing to its small value in a sheet material yields less accurate results.

The R value, is therefore, a useful parameter for indicating the degree of mechanical anisotropy of a given material. For an isotropic sheet, the R value is equal to one. If R is less than one, the sheet thins unduly and is, therefore, undesirable for drawing operations. For deep drawing, it is preferable to have R values equal to or greater than about 1.5; however, for some applications, material with lower R values may be satisfactory.

To obtain an average R value, tensile tests are conducted on several specimens taken at various angles, usually at 0, 45 and 90° to the rolling direction. An average R value of the sheet can then be obtained as 70 follows:

 $\overline{R} = (R_{0^{\circ}} + 2R_{45^{\circ}} + R_{90^{\circ}})/4$

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The difference among the individual R values indicates the earing tendency of the sheet in the drawing operation. The larger the difference, the stronger is the tendency for earing.

It has been found that the crystallographic orientation of the grains, and not the grain shape, is primarily responsible for the drawing properties. We have discovered that the drawability and the R value can be correlated with the crystallographic texture of the sheet. Good drawability and high R values are associated with the socalled "cube-on-corner" or the (111) texture, i.e. the (111) planes are parallel to the plane of the sheet. Poor drawability and low R values are associated with the "cube-on-face" (100) texture. The "cube-on-edge" or the (110) texture has intermediate drawing properties. Thus, for good drawing properties, the amount of the (111) texture should be high, whereas that of the (100) texture should be low. For a specific crystallographic plane, the R value varies also with the directions lying in the ability is (111) fiber texture with the sheet plane normal as the fiber axis.

The crystallographic texture of a specimen is normally determined by the construction of complete pole figures from X-ray intensity measurements; however, for detection of small variation in the texture, a direct comparison of two pole figures cannot reveal the detailed differences quantitatively. Accordingly, we have found it best to measure the integrated peak intensities of several reflections from the plane of the sheet and express them in units of corresponding peak intensities of a random specimen. The numerical values of these relative intensities so obtained are directly proportional to the pole densities of a specific plane lying parallel to the plane of the sheet. Since the drawability of a sheet depends on the relative population of specific crystallographic planes in the plane of the sheet, this technique is very useful. The intensities of five different reflections, i.e. (110), (200), (112), (310) and (222) are measured. The intensity of the (222) reflection which is the second order reflection of the (111) therefore represents the amount of (111) texture. Similarly, the intensity of the (200) reflection represents the amount of the (100) texture, respectively. These two textures have, therefore, effects on drawability 45 as do their counterparts. The correlation between R values and texture has been found to be very consistent in actual test results.

We have found a method of producing low-carbon sheet steel of good drawability without sacrificing yield strength which involves a combination of steps applied to low-carbon steels having initially greater than 0.02% carbon. Our method produces a crystallographic texture with a high degree of (111) orientation and a lesser quantity of (100) orientation. Thus, low-carbon steel processed according to the invention has a very favorable average R value indicating good drawability. In addition, however, we have found that by controlling the final carbon content and precluding undue decarburization, a high yield strength may be maintained and a low-carbon steel having both improved drawability and high strength can be produced. According to the invention, a low carbon, hot rolled plate having more than about 0.02% carbon is cold rolled to from 50% to 85% reduction into sheet gauge. The cold-rolled sheet is then annealed at normal annealing temperature in the range from about 1025° F. to about 1550° F. (below the transformation temperature) for more than 10 hours in an atmosphere containing dry hydrogen. The term "dry hydrogen" used herein refers to hydrogen having a dew point less than -30° F. The sheet steel is annealed at the stated temperature for the stated time until the carbon content is in the range from about 0.004 to about 0.02% and then

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the annealed sheet is cooled. According to the preferred procedure, the hot-rolled plate is cold rolled to from 65 to 75% reduction and annealing is conducted at a temperature in the range 1200 to 1400° F. Annealing is preferably conducted for more than about 15 hours, desirably about 20 hours.

The discussion and examples set out hereinafter will more fully explain the invention. In the ensuing discussion, reference will be made to three continuously-cast, low-carbon steels designated steels "A", "B" and "C" which had the following compositions in percent by

TABLE I.—CHEMICAL COMPOSITION OF STEELS

Steel ·	C	Mn	Si	S	P	Al_{sol}	N	0
A				0.017 0.018				
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It should be noted that compositions A and B had a desired carbon content above 0.02% whereas composition C had a lower initial carbon content. Each of these steels was hot rolled in a commercial mill under the following conditions.

TABLE II.—HOT ROLLING CONDITIONS

Steel	Tem	Finish Thick-		
Buen	Enter	Finish	Coil	ness (in.)
A B C	1, 940–1, 950 1, 940–1, 950 2, 300	1,630 1,630 1,600	1, 150 1, 150 1, 080	0. 096 0. 096 0. 086

The microstructure of the hot-rolled plate from these steels consisted of equiaxed grains of about #8 ASTM grain sizes. In compositions A and B, small colonies of fine pearlite were found to exist between the grains and at three grain junctions and carbide plates were present 35at the grain boundaries. In composition C, pearlite colonies were rare; however, a few thin carbide plates existed at some of the grain boundaries.

Crystallographic examinations (the results of which are reported in Table III below) of the hot rolled plate 40 showed only minor differences between the three steels, except that the (110) intensity of composition C steel was very high and the (222) intensity was lower than in either of compositions A or B. The texture of compositions A and B were practically the same.

TABLE III.—RELATIVE INTENSITIES OF SELECTED X RAY REFLECTIONS FROM HOT-ROLLED PLATE, STEELS A, B, AND C

	A	. В	C
110)	0.80	0, 89	1.12
200)	0.80 1.59	1.70	1.78
12)	1.23	1, 26	1.14
310)	1, 16	1.10	1.10
999)	1.57	1.34	1.14

In aluminum-killed steels, crystallographic texture is 55 controlled by precipitation of aluminum nitride. In lowcarbon steels treated according to the invention, the crystallographic texture is controlled utilizing the cementite normally present. This may be accomplished by various annealing treatments such as the solution and 60 tempering treatments prior to cold rolling, or decarburization during recrystallization anneal to effect the development of desired textures. It has been found that excellent R values can be obtained in low-carbon steels having initial carbon contents of more than about 0.02%, such as compositions A and B, by proper annealing in a dry hydrogen atmosphere whereby the carbon content is reduced to within the range of from 0.004 to 0.02% carbon. It has also been found, as will be shown hereinafter, that the treatment did not produce satisfactory 70 results for composition C because the initial carbon content was already below the 0.02% limit.

Hot-rolled plate samples of compositions A, B and C were cold rolled to 70% reduction in thickness from 0.096 to 0.029 inch for compositions A and B, and from 75 period of the annealing treatment.

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TABLE IV.—RELATIVE INTENSITIES OF SELECTED X-RAY REFLECTIONS FROM DRY HYDROGEN ANNEALED LOW-CARBON STEELS AND A TYPICAL SK STEEL

	A	В	C	SK.
(110)	0. 09 0. 17	0. 17 0. 23	0. 25	0. 25 0. 38
(112)	1.18	1, 12	0.54 1.42	2.04
(222)	0. 24 6. 00	0. 27 5. 80	0.45 4.70	0. 26 4. 90

It can be seen that the unfavorable texture components, i.e. (200) and (110) of compositions A and B are substantially lower than those of the SK grade, whereas the most favorable texture component (222) is higher than that of the SK grade. On the other hand, the (110) and (222) components of composition C are equal to those of the SK grade, but the (200) and (310) components are appreciably higher. The (112) component of all three 25 low-carbon steels are lower than that of the SK steel.

The R values indicating relative drawability and yield strength and grain size, as determined from duplicate samples of these steels which were also tested are shown in Table V. Corresponding data for the SK grade steel are also listed for comparison.

ABLE V.—THE R VALUES, YIELD STRENGTH, AND GRAIN SIZE OF DRY HYDROGEN ANNEALED LOW-CARBON STEELS AS COMPARED WITH THOSE OF A TYPICAL SK STEEL

•		A	В	, C ,	SK
	R ₀ •	1.82	1.77	1. 25	1.73
	R ₄₅ °	1.70		0.91	
	$\overline{\mathbf{R}}_{}$				
	Y.S. (K p.s.i.)	18.6	22.9	31.2	23. 2
	Grain size (ASTM No.)	5.5	6.0	9.0	6.5

As can be seen, the R values of compositions A and B are equal or superior to those of the SK grade steel and the R values for composition C are not as satisfactory. The grains in all three carbon steels, in contrast to the SK grade steels, are equiaxed.

The extent of decarburization depends to some extent on the flow rate of the hydrogen-containing atmosphere and the surface exposure of the specimen. With a flow rate of approximately 60-80 cc./minute, the carbon content may be reduced to as low as 0.004%, if care is exercised in the placement of the sheets. If the sheets are placed loosely in contact with each other, the carbon content can be maintained at about 0.016%. Extensive testing indicates that consistently good texture, high R values and high yield strength are always obtained if the carbon content in the annealed strip is reduced to below 0.02%. On the other hand, we have found that if the final carbon content is greater than 0.02%, the texture and the R values of the annealed strip are invariably poor. The results of numerous tests indicate that during the dry hydrogen anneal, the desired crystallographic texture is developed through the influence of the iron carbide precipitate on the growth characteristics of the grains.

The effect of heating rate and soaking time on plastic strain ratio is shown in Table VI. These results show that at a constant soaking time the average R values decrease slightly with increasing heating rate and with a constant heating rate, R decreases with decreasing soaking time. The remaining carbon content correlates with the R values and shows that poor R values are associated with high carbon content, i.e. carbon contents greater than 0.02%. The remaining carbon contents indicate also that the effective decarburization occurs mainly during the soaking

TABLE VI.—EFFECT OF HEATING RATE OR SOAKING TIME ON THE AVERAGE PLASTIC STRAIN RATIO, \overline{R} , AND THE REMAINING CARBON CONTENTS

Heating rate_	Soaking time	$\overline{\mathbf{R}}$		C, percent		5	
(time to 1,320° F.)	(Hr. at 1,320° F.)	A	В	A	В	Ü	
6 min	20 20 20 20 20 2 10 20	1. 65 1. 75 1. 74 1. 97 1. 15 1. 35 1. 97	1. 45 1. 59 1. 53 1. 60 1. 26 1. 39 1. 60	0. 011 0. 011 0. 007 0. 029 0. 024 0. 007	0. 011 0. 010 0. 004 0. 034 0. 023 0. 004	10	

To illustrate the importance of using dry hydrogen, a series of tests were conducted which differed only in that in some dry hydrogen was employed and in others wet hydrogen was used. Although, as is known, wet hydrogen is more effective for decarburization than dry hydrogen, we have found that excessive decarburization and grain growth results with the use of wet hydrogen even though acceptable R values may be obtained. Table VII reports the results of four tests of compositions A and B, two of which were conducted with the annealing performed in dry hydrogen and two with the annealing in wet hydrogen. 25 The results of these tests are reproduced in Table VII.

TABLE VII

	Dry H	drogen	Wet Hydrogen		
•	A	В	A	В	
R ₀ o	1,90	1, 68	1.82	1,54	
R ₄₅ °	1.76	$1.21 \\ 2.26$	$1.43 \\ 2.25$	1. 20 2. 08	
R ₀₀	2.55 1.99	1.59	1.73	1.50	
VS (Knsi)	1, 99 17, 8	23. 2	10.0	9.8	
Grain size (ASTM No.)	6.0	7.0	6-1	7.0	
Carbon Content	0.007	0.004	. 0015	.00150014	

It is apparent from the results shown in Table VII that, as indicated above, although the R values obtained by annealing in wet hydrogen are generally satisfactory, the yield strength is only about half of that obtained by annealing in dry hydrogen. This difference may be due to the low residual carbon content and/or the tendency for larger grains which results from annealing in wet hydrogen.

The chemical composition of the dry hydrogen annealed specimens A and B is shown below in Table VIII. As can be seen, the nitrogen content has been lowered significantly. Since unfixed nitrogen is a principal cause of strain aging of low-carbon steels, the undesirable effects of aging on mechanical properties can be minmized by this treatment.

TABLE VIII.—CHEMICAL COMPOSITION OF STEEL AFTER DRY HYDROGEN ANNEAL

	Steel	C	Si	S	N	0
A		0.007	0. 02	0. 016	0.001	0.009
B		0.004	0. 03	0. 016	0.001	0.008

The strain aging index of dry hydrogen annealed products is shown below in Table IX.

TABLE IX.—Strain aging index 1 of dry hydrogen annealed steels (prestrained 8%, aged 4 hrs. at 212° F., and retested)

Percent increase in yield stress (Strain Aging Index):

Α	 12.7
	 40 4
В	 12.4

¹The percent increase in yield stress when the specimen is prestrained 5% minimum or beyond discontinuous yielding, aged 4 hours at 212° F., then retested.

The above values compare favorably to the strain aging index of conventional box-annealed rimmed steels, which usually ranges between 20 to 25%.

It is apparent from the above that certain changes and modifications may be made without departing from the invention. It is seen, however, that it is essential for the successful practice according to the invention for the inital carbon content of the steels treated to be above 0.02% and, further, that the carbon be reduced during the dry hydrogen anneal to from between 0.016 to 0.004%. The effect of this treatment is to impart improved drawability without sacrificing yield strength. It is also shown that too extensive decarburization results in excessive grain growth which impairs the drawability and yield strength of the sheet steel. Moreover, the product is relatively nonaging as well as deep drawable due to the fact that the dry hydrogen anneal also reduces the nitrogen content considerably, i.e., to about 0.001%.

We claim:

- 1. A method of producing low-carbon sheet steel of improved drawability and high yield strength which comprises cold rolling hot-rolled plate of low-carbon steel having more than 0.02% carbon to from 50 to 85% reduction into sheet gauge, annealing said cold-rolled sheet at a temperature in the range of from about 1025° F. to about 1550° F. for more than ten hours in an atmosphere containing dry hydrogen having a dew point less than -30° F, to result in a carbon content of from 0.004 to less than 0.02% and cooling the annealed sheet.
- 2. A method according to claim 1 wherein said hot-40 rolled sheet is cold rolled to from 65 to 75% reduction.
 - 3. A method according to claim 1 wherein said cold-rolled sheet is annealed at a temperature in the range of 1200° F. to 1400° F.
- 4. A method according to claim 3 wherein said cold-45 rolled sheet is annealed for more than 15 hours.
 - 5. A method according to claim 4 wherein said cold-rolled sheet is annealed for about 20 hours.

References Cited

UNITED STATES PATENTS

2,360,868	10/1944	Gensamer 148—16
3,239,388	3/1966	Sasaki 148—12.1
3,239,389	3/1966	Yoshida 148—12.1
3,244,565	4/1966	Mayer et al 148—12.1
3,262,821	7/1966	Yoshida 148—12.1
3,281,286	10/1966	Shimizu et al 148—16
3,348,980	10/1967	Enrietto 148—12.1

OTHER REFERENCES

Low et al., vol. 158, AIME Transactions, Iron and Steel Division, p. 209 et seq.

HYLAND BIZOT, Primary Examiner.

W. W. STALLARD, Assistant Examiner.