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TREATMENT OF SHEET STEEL

Edward H. Mayer, Bethlehem, Pa., assignor, by mesne assignments, to Bethlehem Steel Corporation, a corporation of Delaware

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This application is a continuation-in-part of application Serial No. 94,721, filed March 10, 1961, now abandoned.

This invention relates to the treatment of steel, and more particularly to an improved method for treating steel intended for deep drawing.

A principal object of this invention is to provide an improved method of treating steel sheet to prevent brittleness in said sheet, thus improving the deep drawing qualities thereof.

Another object is to provide a method of treating decarburized sheet steel to improve its deep drawing qualities.

In one prior art method of decarburizing steel, steel strip of ordinary rimming steel composition containing for example 0.08% C and 0.40% Mn is wound into an open coil and heated to a temperature of approximately 1100° F. in a dry, non-oxidizing atmosphere. Upon reaching this temperature, the strip is annealed at a temperature between 1100°–1400° F. in a gas of approximately the following composition:

Hydrogen (H_2)	18%.
Water (H_2O)	Dewpoint +95° F. (5.5 vol. percent).
Carbon oxides (CO and CO_2)	Less than 0.5%.
Nitrogen (N_2)	Balance.

This gas is believed to decarburize the steel in the manner shown by the following reaction:

$$H_2O \text{ (in gas)} + C \text{ (in steel)} \rightarrow CO + H_2$$

This treatment is continued until the carbon in the steel has been reduced to the desired level, say below 0.01%. The steel is then cooled to ambient temperature in a dry reducing atmosphere.

The decarburized steel will, ordinarily, have the ductility requisite for deep drawing operations. However, in the production of deep drawing steel by this method, rejects may occur as a result of brittle fracture of the decarburized product during the forming operation.

This type of fracture generally occurs along the grain boundaries, and is known as a brittle fracture, and is to be distinguished from the type of failure generally associated with deep drawing steel, annealed by conventional methods such as box annealing, known as ductile failure, which occurs when the steel necks down in a local area.

It is extremely difficult, if not impossible, to distinguish the brittle stock from ductile stock, by the usual tests such as Olsen cup, sheet tensile and hardness tests, applied to the stock after decarburizing. For this reason, the brittle characteristics of the stock are discovered only after the drawing fracture has occurred.

Embrittlement, as the term is used herein, refers to that condition wherein a steel exhibits a degree of brittle fracture which precludes use of the steel for deep drawing operations.

I have found that if the decarburizing treatment in wet hydrogen is followed by a further treatment in a dry hydrogen atmosphere, for a critical minimum time period and within a critical temperature range, brittleness is eliminated to a point where it is no longer a problem in subsequent forming operations.

In one example of the method by which my invention can be performed, a 56 inch width, 20-gage coil of cold rolled strip, of the composition above mentioned, and

weighing approximately 10 tons, is wound into an open coil, by inserting a spacer between adjacent laps to permit the furnace gases to contact both surfaces of the strip. The open wound coil is introduced into a decarburizing furnace (covered pot annealing furnace), and heated to 1100° F. in an atmosphere of about 4% H_2 and 96% N_2, having a dewpoint of −40° F. (0.01% H_2O).

When the furnace attains a temperature of 1100° F., the dry gas in the furnace is replaced with a decarburizing atmosphere. The decarburizing gas is a wet gas having a +95° F. dewpoint (5.5% H_2O) and containing 18% H_2 (dry basis) and 82% N_2 (dry basis). The furnace temperature is increased to 1290° F., and held at this temperature until the decarburization has been carried to the desired point, say to a carbon content of 0.003% or lower. Completion of the reaction is determined by the results of a test for carbon monoxide made upon the furnace exit gas.

Upon completion of the decarburization reaction, the furnace is purged of the wet decarburizing gas, and a dry gas (−40° F. dewpoint) containing about 82% N_2 and 18% H_2 is introduced into the furnace. The steel coil is held at about 1290° F. in the dry reducing atmosphere for a period of 24 hours.

At the end of the dry gas soak, or holding period, the furnace gas is again changed, this time a dry (−40° F. dewpoint) gas containing about 4% H_2 and 96% N_2 replaces the soaking gas. The furnace is then cooled to 250° F. and uncapped.

Steel coils, treated by my combination decarburizing-soaking method, are skin-rolled by conventional methods, and are then ready for shearing and deep drawing. The treated steel now has forming characteristics which permit it to be subjected to severe deep drawing operations with no significant brittle fracture failure.

For the purpose of this invention, the dry gas soak may follow any steel decarburization operation as above described in which there is present sufficient moisture, within certain limits, to cause embrittlement of the steel. The maximum moisture content is determined by the H_2O/H_2 ratio above which scaling of the steel surface will occur. This H_2O/H_2 ratio is approximately 0.32 at 1100° F. and 0.49 at 1400° F.

It is to be understood that while the process just described includes operating conditions of temperature, time and gas content found desirable in producing a satisfactory non-brittle decarburized steel, the invention is by no means limited to these conditions.

Laboratory work shows that with a dry gas containing 18% H_2, at a temperature of 1290° F., the holding time may be reduced to 3 hours. Other laboratory work shows that with a temperature of 1290° F. and a holding period of 24 hours, the H_2 content may be reduced to 4%. Thus the gas composition and holding time specified in the above example represent a liberal safety factor for satisfactory commercial practice. With a soaking gas such as that described in the example, it is possible to reduce the holding time to as little as 3 hours. Even less than 3 hours' holding time may be feasible with a dry gas of appreciably higher hydrogen content. An atmosphere containing as much as 100% H_2 (dry basis) could be used in soaking, although such an amount would not be economical.

Variations in the moisture content of the gas in the decarburizing step will affect the duration of the subsequent dry gas soak. For example, if the dewpoint of the decarburizing gas is lowered to +70° F. (2.47% H_2O) a somewhat shorter time period is required for the dry gas or soaking treatment.

In the heating up and cooling cycles, considerable latitude as to the nature and quantities of ingredients of the atmosphere is permissible, the particular composition described here being one normally used in annealing operations to protect the steel from scaling.

A desirable temperature range for both the decarburization and dry gas treatments is between 1100° F. to 1400° F. Above 1100 F. the reaction and carbon diffusion rates are sufficiently rapid to complete the decarburization in a reasonable length of time. At lower temperatures, these rates would be decreased to a point where the process would take an unduly long time to complete the reactions. Furthermore, at temperatures below 1100° F., the cold worked steel would not recrystallize during the decarburizing treatment, and thus would not be in the soft, ductile condition required for the finished product. A temperature of 1400° F. is approximately the maximum at which the desired grain size can be obtained to give satisfactory drawability, although in most cases it is preferable to operate at 1290° F. or slightly below. Higher decarburizing and dry treatment temperatures result in excessive grain coarsening, which in turn leads to objectionable "orange peel" on deep drawn parts. Excessive softening also results when the grain is too coarse.

While I do not wish to be held to any specific theory by which steel stock, treated by the process of my invention, is relieved of embrittlement, a preponderance of data resulting from tests made on deep-drawing steel stock treated by my process, as well as from tests made on similar stock untreated by my process, lead to certain apparently valid conclusions.

It has been observed that the microstructure of brittle decarburized steel exhibits oxides beneath the surface. Some of these oxides are distributed at the grain boundaries, while others are within the body of the grain. Only those grains near the surface exhibit oxides visible with 500× magnification. The oxides penetrate to a depth of from 0.001 in. to 0.002 in. below the metal surface. It has been established by electron diffraction studies, that the oxides are ferrous-ferric oxide (Fe_3O_4).

Photomicrographs made at 500× magnification, from specimens taken from steel stock which has been decarburized by the method of the present invention, show that the grain boundary oxides have been reduced to a considerable degree. Apparently, oxygen at the grain boundaries is the cause of embrittlement in steel stock which has been decarburized without the benefit of a dry gas soak, and the partial elimination of this oxygen by the dry gas treatment of this invention restores the normal ductility to the steel.

The degree of dryness required for the gas in the soaking step may be dictated by convenience, within a relatively wide dewpoint range.

In the example given above, the dry gas may have a dewpoint of up to about +20° F. (0.34% H_2O) to +25° F. (0.44% H_2O).

In a commercial process, water vapor, which is formed from the oxygen removed from the steel, mixes with the circulating dry gas in the soaking operaiton, thus raising the dewpoint of the soaking gas. This additional moisture should be taken into account when introducing the dry gas to the system, so that the dry soaking gas is maintained, during the entire soaking operation, at a dewpoint of +20° F. to +25° F. or lower.

The holding time, during which the decarburized steel is held in the dry gas soak, refers to that period during which is is possible for effective deoxidation of the steel to take place, for, if the dry gas soak follows immediately the decarburizing step, the high dewpoint decarburizing atmosphere must be flushed from the system before the dry soaking gas becomes initially effective.

I have found that the oxygen content of decarburized steel is a measure of its susceptibility to brittle fracture on forming and that the danger of brittle fracture is eliminated if the oxygen content of the material is not greater than about 0.020%. By way of example, a sample of sheet steel decarburized in wet hydrogen by conventional methods was found to have an oxygen content of 0.025% and to exhibit brittle fracture on forming. After the sample was held in dry hydrogen at 1300° F. for 24 hours, it contained 0.020% oxygen, and on forming, was not subject to brittle failure. Thus we may establish a relationship between the presence of grain boundary oxidation and brittle fracture.

The method of this invention is especially useful in treating enameling steel of the type described in United States Patent No. 2,956,906 to Blickwede et al.

I claim:

1. The method of treating steel which comprises holding the steel in a decarburizing atmosphere comprising hydrogen and water vapor as the essential active ingredients at a temperature between 1100° F. and 1400° F. until decarburization is substantially complete, changing the atmosphere to a dry gas containing not less than about 4% hydrogen as the essential active ingredient and holding the steel in said dry gas for not less than 3 hours at between 1100° F. and 1400° F., then cooling the steel in a protective atmosphere.

2. The method of treating sheet steel which comprises heating said steel to at least 1100° F. in a dry nonoxidizing atmosphere, changing the atmosphere to a decarburizing gas comprising hydrogen and water vapor as the essential active ingredients, maintaining the temperature at between 1100° F. and 1400° F. until decarburization is substantially complete, again changing the atmosphere to a dry gas containing not less than about 4% hydrogen as the essential active ingredient and holding the steel in said second dry gas for not less than 3 hours at between 1100° F. and 1400° F., then cooling the steel in a dry non-oxidizing atmosphere.

3. The method of treating sheet steel which comprises heating said steel to at least 1100° F. in an atmosphere of dry gas containing at least 4% H_2 and the balance substantially nitrogen, changing the atmosphere to a decarburizing gas containing about 18% H_2 and the balance substantially nitrogen calculated on a dry basis and having water vapor equivalent at least to a +32° F. dewpoint, increasing the temperature to about 1290° F. and maintaining the steel at the increased temperature until decarburization is substantially complete, again changing the atmosphere to a dry gas containing about 18% H_2 and 82% N_2 and holding the steel in said second dry gas for about 24 hours at about 1290° F., then cooling the steel in a dry gas containing about 4% H_2 and 96% N_2.

4. The method of treating low-carbon rolled steel stock which comprises holding the stock in a decarburizing atmosphere comprising hydrogen and water vapor as the active ingredients at a temperature between 1100° F. and 1300° F. until the stock is decarburized below 0.01% carbon, changing the atmosphere to a dry gas containing not less than about 4% hydrogen as the essential active ingredient and holding the stock in said dry gas for not less than 6 hours at between 1100° F. and 1350° F., then cooling the steel in a protective atmosphere.

5. The method of treating rolled steel stock which comprises holding the stock in a decarburizing atmosphere comprising hydrogen and water vapor as the active ingredients at a temperature between 1100° F. and 1400° F. until decarburization is substantially complete, said water vapor being present in sufficient amount to effect decarburization relatively rapidly but in insufficient quantity to cause scaling of the steel stock surface, changing the atmosphere to a dry gas containing not less than about 4% hydrogen as the essential active ingredient and holding the stock in said dry gas for not less than 3 hours at between 1100° F. and 1400° F., then cooling the stock in a protective atmosphere.

6. The method of treating steel which comprises holding the steel in a decarburizing atmosphere comprising hydrogen and water vapor as the essential active ingredients at a temperature between 1100° F. and 1400° F. until decarburization is substantially complete, changing

the atmosphere to a dry gas containing not less than 4% hydrogen and the balance a neutral gas and holding the steel in said dry atmosphere for not less than 3 hours at between 1100° F. and 1400° F., then cooling the steel in a protective atmosphere.

7. The method of treating steel which comprises holding the steel in a decarburizing atmosphere comprising water vapor and hydrogen as the active ingredients at a temperature between 1100° F. and 1400° F. until decarburization is substantially complete, the water vapor and hydrogen having an H_2O/H_2 ratio not greater than 0.49 at 1400° F., changing the atmosphere to a dry gas containing not less than about 4% hydrogen as the essential active ingredient and holding the steel in said dry gas for not less than 3 hours at between 1100° F. and 1400° F., then cooling the steel in a protective atmosphere.

8. The method of treating steel which comprises holding the steel in a decarburizing atmosphere comprising hydrogen and water vapor as the essential active ingredients at a temperature between 1100° F. and 1400° F. until decarburization is substantially complete, changing the atmosphere to a dry gas containing not less than 4% hydrogen and the balance a neutral gas and holding the steel in said dry atmosphere at between 1100° F. and 1400° F. for a period of time sufficient to relieve the steel of embrittlement, then cooling the steel in a protective atmosphere.

9. The method of treating sheet steel which comprises holding decarburized sheet steel containing at least 0.025% oxygen at a temperature between 1100° F. and 1400° F. in a dry atmosphere containing not less than about 4% hydrogen as the essential active ingredient for not less than 3 hours and cooling the steel in a protective atmosphere.

10. The method of treating sheet steel which comprises holding decarburized sheet steel containing less than 0.01% carbon and at least 0.025% oxygen to a temperature between 1100° F. and 1400° F. in a dry atmosphere containing not less than about 4% hydrogen as the essential active ingredient for not less than 3 hours and cooling the steel in a protective atmosphere.

11. The method of treating sheet steel suitable for deep drawing and free from brittle failure on forming which comprises holding the steel in a decarburizing atmosphere comprising hydrogen and water vapor as the active ingredients at a temperature between 1100° F. and 1300° F. to reduce the carbon content of the steel to a desired level and simultaneously produce a material having an oxygen content in excess of 0.020%, and thereafter holding the steel in a dry atmosphere containing not less than about 4% hydrogen as its essential ingredient, at a temperature between 1100° F. and 1400° F. for a period of time sufficient to reduce the oxygen content of the steel to a maximum of 0.020%.

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BENJAMIN HENKIN, *Primary Examiner.*

DAVID L. RECK, *Examiner.*