United States Patent [19]

Suzuki et al.

[11] **3,865,636**

[45] **Feb. 11, 1975**

[54] **METHOD OF PROCESSING STEEL MATERIAL HAVING HIGH AUSTENITIC GRAIN-COARSENING TEMPERATURE**

[75] Inventors: **Akira Suzuki**, Kobe; **Shushi Kinoshita; Takeshi Ueda**, both of Akashi, all of Japan

[73] Assignee: **Kobe Steel, Limited**, Japan

[22] Filed: **July 18, 1973**

[21] Appl. No.: **380,460**

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 134,332, April 15, 1971, Pat. No. 3,788,903.

[30] **Foreign Application Priority Data**

Apr. 15, 1970 Japan 45-32078

[52] **U.S. Cl.** ..... **148/12 R**, 148/12 F, 148/12.1

[51] **Int. Cl.** **C21d 7/02**, C21d 7/14

[58] **Field of Search** 148/12, 12.1

[56] **References Cited**

UNITED STATES PATENTS

3,711,342 1/1973 Cullen et al. 148/12

Primary Examiner—W. Stallard

Attorney, Agent, or Firm—Oblon, Fisher, Spivak, McClelland & Maier

[57] **ABSTRACT**

A method of processing steel material having a high austenitic grain-coarsening temperature and an abnormal grain growth curve of cold worked austenitic grain, which comprises heating said steel material at a temperature of from the ferrite recrystallization temperature to the A_1 transformation point for more than five minutes to cause recrystallization. This technique prevents the formation of mixed grains upon austenitizing and thereby preserves the mechanical properties of the steel material.

8 Claims, 4 Drawing Figures

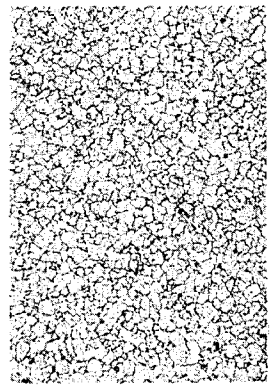


FIG. 1A

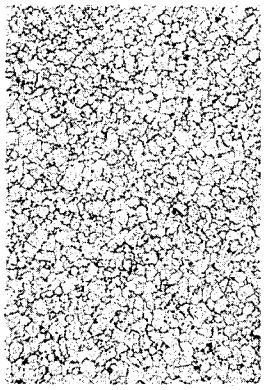


FIG. 1B

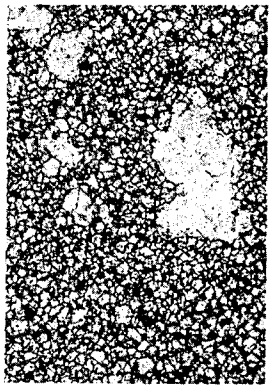


FIG. 2A

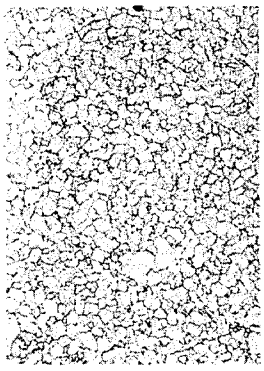
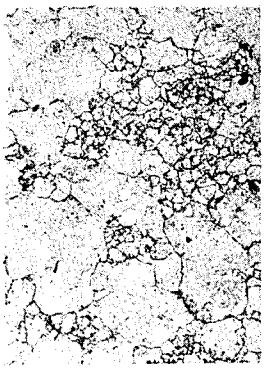


FIG. 2B



METHOD OF PROCESSING STEEL MATERIAL HAVING HIGH AUSTENITIC GRAIN-COARSENING TEMPERATURE

RELATIONSHIP TO RELATED APPLICATIONS

This application is a continuation-in-part of copending application, Ser. No. 134,332, filed Apr. 15, 1971, now U.S. Pat. No. 3,788,903.

BACKGROUND OF THE INVENTION

1. Field Of The Invention

This invention relates to a method of processing steel material having a high austenitic grain-coarsening temperature, and more particularly to a method of processing steel material without the danger of producing a mixed grain structure upon heat treatment in the austenitic region following cold working.

2. Description Of The Prior Art

If carbon steel and alloy steel materials containing elements having grain refining action, such as aluminum, titanium, niobium, etc., are heated in their austenitic region, they show an abnormal grain growth curve. When steel containing a grain refining element is cold worked at a temperature lower than the ferritic recrystallization temperature, the temperature at which austenitic grains on subsequent austenitization will start to abnormally grow, will be depressed, and will result in a mixed grain structure.

The mixed grained steels are usually considered to be of inferior quality, and, when such steels are heat treated under severe austenitizing conditions, such as carburizing in the austenitic region following cold working, difficulties with toughness, strain and distortion can result.

Heretofore, it has been found that heating the steel at a temperature above 1,000°C. after cold working, will reduce the dangers of producing a mixed grain structure since the austenitic grain-coarsening temperature of the cold worked steel will be increased. However, such high temperature heat treatment can cause serious scale formation which can result in severe dimensional inaccuracy and can impair the smooth surface created by cold forming. Moreover, such high temperature heat treatment is not economically desirable from a commercial cost standpoint.

SUMMARY OF THE INVENTION

Accordingly, it is one object of the present invention to provide a method of processing steel which improves the depressed austenitic grain-coarsening temperature caused by cold working, and minimizes the danger of formation of mixed austenitic grain structure upon subsequent austenitizing.

It is another object of this invention to prevent the deterioration of toughness, strain and distortion of steel material due to the presence of a mixed austenitic grain structure.

Another object of the present invention is to provide a method of processing a steel material without losing the desirable attributes of cold working, including its excellent dimensional accuracy, smooth surface skin, etc.

A still further object of the present invention is to provide a method of processing a steel material which does not create mixed grains during carburizing treatment following cold working in the preparation of machine elements.

These and other objects have now herein been attained and the austenitic grain-coarsening temperature increased by recrystallizing the cold worked ferritic structure at a temperature within the ferritic recrystallization region, below A_1 transformation temperature, before the austenitizing heat treatment. It is believed that this technique reduces the driving force of the austenitic grain growth upon heat treatment of the steel material.

BRIEF DESCRIPTION OF THE DRAWINGS

A more complete appreciation of the invention will be readily obtained with the aid of the following description in conjunction with the accompanying Tables and photographs.

FIGS. 1(*a*) and 2(*a*) are photographs (×100) showing the preferable fine grain structure obtained after austenitizing the steel material at 950°C. by the method of this invention; and

FIGS. 1(*b*) and 2(*b*) are photographs (×100) showing mixed grain structure appearing after austenitizing the same steel material at 950°C. by the conventional method.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS

Any steel material which is characterized by abnormal austenitic grain growth, i.e., any steel material in which the austenitic grains start to grow suddenly at a temperature in the austenitic region, can be treated according to the present invention. For instance, this technique can be successfully applied to carbon steels and alloy steels containing one or more grain refining elements, such as aluminum, titanium, niobium, zirconium, vanadium, tantalum, rare earth elements, etc. which precipitate as nitrides, carbides and oxides.

The steel material is prepared for cold working, after hot working and/or heat treatment, etc., and is formed to a desired shape by rolling, forging, extruding, etc., at a temperature below the ferritic recrystallization temperature region. Mechanical working, such as machining, may be used as the cold working.

The cold worked steel material has a lower austenitic grain-coarsening temperature than non-cold worked steel material as aforementioned. This steel material is then recrystallized at a temperature within the ferritic recrystallization region so as to raise the austenitic grain-coarsening temperature and to thereby cause the cold worked ferrite structure of the steel material to substantially recrystallize in the ferritic region. To accomplish this, the steel material must be heated to at least the temperature within the ferritic recrystallization range, but below the A_1 transformation temperature. The specific temperature range will, of course, depend upon the type of steel and the cold working conditions, but the lower limit is usually about 400° – 500°C., while the upper limit is about 700° – 800°C.

If the temperature of the heat treatment is lower than the lower limit, the deformed ferritic structure will not substantially recrystallize, while if the temperature of the heat treatment is higher than the upper limit, the deformed ferritic structure will not be recrystallized and it will result in lower austenitic grain-coarsening temperature with its consequent risk of mixed grain structure formation on subsequent austenitizing. In general, within the desired temperature range, the

lower the heating temperature, the longer the period of heating time required. The shortest time period is 5 minutes, but periods of longer than 30 minutes are preferred. When the heat time period is less than 5 minutes, sufficient recrystallization will not occur.

One good method for heat treating is to hold the steel material at a constant temperature within the ferritic recrystallization region, and thereafter to cool it. However, the same effect may be expected by cyclically heating and cooling within the ferritic recrystallization region at a sufficient cyclic rate.

The heat treatment in the ferritic recrystallization region according to this invention will be effective regardless of whether the austenitic grain growth inhibitors are nitrides, carbides or oxides and regardless of whether they have already precipitated before or are in the process of precipitating before the ferritic recrystallization treatment. It is also effective, regardless of the state of the precipitation, and regardless of the amount, number, distribution and/or morphology of the particles. However, the positive utilization or progression of the precipitation during the ferritic recrystallization treatment will further raise the austenitic grain-coarsening temperature, since it results in finer austenitic grain growth inhibitor particle size. Another characteristic feature of this invention, therefore, is that the combination of increased inhibiting force against grain-coarsening and reduced grain growth driving force on subsequent austenization will raise the austenitic grain-coarsening temperature. The increased inhibiting force is derived from the precipitation treatment of the supersaturated steel materials in the ferritic region, and the reduced grain growth driving force is a result of the ferritic recrystallization treatment following cold working.

Since it is necessary to dissociate the nitrides and carbides into a solid solution, during the solution treatment of steel materials, it is necessary to heat the steel to a temperature of at least 1,000°C. for an excess of 5 minutes. If the heating is below 1,000°C. or for shorter periods than 5 minutes, the dissociation of the precipitate will not be sufficient and the sufficient amounts of fine particles necessary to prevent austenitic grain-coarsening at lower austenitic temperatures on subsequent austenitization, may not be obtained during the ferritic precipitation treatment.

The cooling rate after solution treatment should be not less than 5°c./min. in the temperature range of 900° to 500°C. Cooling rates of less than 5°C./min. give rise to the precipitation of a considerable amount of coarse precipitate, which is less effective in the prevention of austenitic grain-coarsening on subsequent austenitization, and gives rise to a reduced amount of finely precipitated particles in the following precipitation treatment. Finely precipitated particles are more effective in inhibiting the austenitic grain-coarsening.

Hot working, such as rolling, forging and extruding may follow the solution treatment to impart cold work performance to the steel material. Cooling should then follow the hot work.

The steel material is in a supersaturated solid solution with nitrogen, carbon and/or substitutional alloy elements to form nitrides and/or carbides after cooling at a definite rate, following solution treatment (and/or hot working). The supersaturated steel material is subjected to precipitation treatment so that the precipitated particles act as the austenitic grain-coarsening inhibitor on the succeeding austenization. This increases the austenitic grain-coarsening temperature. Heating of the supersaturated steel material is most preferable in the temperature range of 500°C. to the A_1 temperature for more 5 min. The precipitate forms as fine particles in the ferritic structure and the rate of precipitation will depend upon temperature. Below 500°C., the rate of the precipitation is so slow that there is substantially no precipitation progress. On the other hand, the precipitation treatment in the austenitic region results in coarser precipitate particles which are not desirable. Heretofore, it was quite common to apply a normalizing treatment prior to cold working. Normalizing should not be conducted as the heat treatment, however, before cold working in this invention.

Heat treatment of the steel material in the higher ferritic temperature region before cold working reduces the deformation resistance upon cold working, and raises the critical working rate above that at which cold working cracks are generated. If more severe cold working performance is required, this treatment may be combined with a spheroidizing annealing in order to improve cold workability.

Austenitizing by intermediate heat treatment, such as annealing which may be carried out between cold working processes, should also be avoided, in order to inhibit austenitic grain-coarsening.

As will be clearly described in the embodiment of the present invention, when the techniques of this invention are applied to cold worked steel materials, the austenitic grain-coarsening temperature will be raised, and thus the dangers of mixed grain structure formation upon austenitization with its consequent deterioration of quality is minimized. Since the method of this invention does not necessitate high temperature treatment, compared with conventional methods for achieving similar results, it can preserve the excelllent dimensional accuracy and smooth surface skin advantages of cold working. Furthermore, the method of this invention is particularly effective when applied to the steel material for machine elements which are subjected to severe austenitizing conditions, such as carburizing treatment, after cold working.

The present invention will be described with reference to the photographs FIGS. 1 and 2 together with certain preferred embodiments.

Table 1 shows the chemical composition of test materials adopted for the embodiment of this invention and the comparison data thereof.

TABLE 1: Chemical Composition Of Test Materials

(Unit: Wt.%)

Contents Type of Steel	C	Si	Mn	P	S	Cr	Mo	Ti	Nb	Sol Al
A	0.20	0.22	0.37	0.005	0.010					0.025
B	0.18	0.27	0.46	0.006	0.009	1.01	0.20			0.036

TABLE 1: Chemical Composition Of Test Materials —Continued

(Unit: Wt.%)

Contents Type of Steel	C	Si	Mn	P	S	Cr	Mo	Ti	Nb	Sol Al
C	0.17	0.28	0.55	0.007	0.012	0.98	0.22	0.025		
D	0.19	0.23	0.41	0.006	0.011	1.04				0.036
E	0.10	0.23	0.40	0.005	0.010	1.01				0.035
F	0.22	0.22	0.40	0.005	0.010	1.03	0.30		0.38	
G	0.20	0.26	0.45	0.005	0.008	0.99	0.15	0.045		

In Table 1, steel A contains aluminum needed to normal carbon steel. Steel B is Cr—Mo steel containing aluminum. Steels C and G are Cr—Mo steels containing titanium. Steels D and E are Cr-steel, containing aluminum. Steel F is Cr—Mo steel, containing niobium.

Table 2 shows the austenitic grain-coarsening temperature measured after holding for 1 hour at the temperatures of ferritic recrystallization region and below it, followed by austenitizing of the steel materials A, B and F, produced by the following process:

1,100°C. X1 hr. solution treatment → 50% hot work ——→ air cooling (average cooling rate: 20°C/min.) ——→ 50% cold working.

"Room Temperature" in Table 2 means that austenitizing was conducted directly after cold working. In Table 2, the increase of austenitic grain-coarsening temperature of the steel material is recognized in the ferritic recrystallization temperatures at and above 450°C.

Table 3 shows the austenitic grain-coarsening temperatures in relation to the heating time at 700°C. for ferritic recrystallization, for steel B produced in the same process as that for Table 2.

TABLE 3

Holding Temperature for Ferritic Recrystallization And Austenitic Grain-Coarsening Temperature

Type of Steel	700° Holding Time	5	20	60	300	800° Holding Time	5	20	60	300
A		925	950	950	975		900	925	925	950
B		950	950	975	1000		925	950	950	950
F		950	975	1000	1000		925	950	950	950

NOTE:
Steel A: hot working — normalizing — cold working
Steel B: hot working — annealing — cold working
Steel F: hot working — spheroidizing — cold working

As is clear from Table 3, the increase of the austenitic grain-coarsening temperature is evident for holding times of 5 minutes or more at 700°C.

The uniform and fine microstructure was obtained by

TABLE 2

Holding Temperature For Ferritic Recrystallization And Austenitic Grain-Coarsening Temperature

Type of Steel	Holding Temp.(°C) Transformation Temp. (°C) A_1	A_3	Room Temp.	Holding Temp. After Cold Working 400	450	500	600	650	700	725	750	775	800	825	850	900
A	730	840	900	900	910	925	925	925	950	950	950	950	925	925	900	900
B	740	845	925	925	940	950	950	975	975	1000	975	975	950	950	925	900
F	740	845	925				950	950	975	1000	1000	1000	950	950	925	925

NOTE:
Steel A: hot working — normalizing — cold working
Steel B: hot working — annealing — cold working
Steel F: hot working — spheoridizing — cold working
1100°C. X 1 hr. solution treatment → 50% hot working
→ air cooling (average cooling rate 20° C/min)
→ 50% cold working.

the combined heat treatment of (1) holding at 700°C. for 1 hour followed by (2) air cooling and austenitizing at 950°C. (followed by quenching) as shown in FIG. 1(*a*) for steel B which was produced by the process indicated in the affix of Table 2. On the other hand, the mixed grain structure was obtained after austenitizing at 950°C without heating at the ferritic recrystallization temperature of 700°C.

A uniform and fine structure, similar to that in FIG. 2(*a*), was obtained for steel C by the same treatment as for FIG. 1(*a*), as shown in FIG. 2(*a*). On the other hand, a mixed grain structure was obtained for steel C treated in the same way as for FIG. 1(*b*), as shown in FIG. 2(*b*).

Table 4 shows the comparison between the austenitic grain-coarsening temperatures of steel D treated by the process of the invention:

solution treatment → cooling → cold working → ferritic recrystallization

or

solution treatment → cooling → AlN fine precipitation treatment → cold working → ferritic recrystallization, and by the conventional treatment:

solution treatment → cooling → normalizing → cold working.

TABLE 4

Treating Process And Austenitic Grain-Coarsening Temperature		
Type of Steel: D		
		Coarsening Temp. (°C.)
This Invention	*1100°C. X 1 hr. solution treatment → 700°C. X 1 hr.AC → 50% cold working → 700°C. X 1 hr. AC	975
	*1100°C. X 1 hr. solution treatment → → 900°C. X 1 hr.AC → 50% cold working	950
Conventional Treatment	*1100°C. X 1 hr.→ 900°C. X 1 hr.AC → 50% cold working	900

"*" indicates mean cooling speed after solution treatment 8°C. min.

Table 5 shows the comparison between the austenitic grain-coarsening temperature of the steel E treated by the process of the invention:

solution treatment → hot working → cooling →AlN fine precipitation treatment → cold working → (intermediate heat treatment → cold working) → ferritic recrystallization, or

solution treatment → hot working → slower cooling → AlN fine precipitation treatment (slightly) → cold working → ferritic recrystallization, and the conventional process:

solution treatment → hot working → cooling → normalizing → cold working.

TABLE 5

Treating Process And Austenitic Grain-Coarsening Temperature		
(Type of Steel: E)		
	Process	Coarsening Temp. (°C.)
This Invention	*1100°C. X 1 hr. solution treatment → hot working* → 500°C. X 50 hrs.AC → 50% cold working → 500°C. X 30 hrs.AC	925
	1100°C. X 1 hr. solution treatment → hot working → 500°C. X 1 hr.AC → 50% cold working → 700°C. X 30 min.AC	950
	1100°C. X 1 hr. solution treatment → hot working → 700°C. X 10 min.AC → 50% cold working → 700°C. X 10 min.AC	925
	1100°C. X 1 hr. solution treatment → hot working → 700°C. X 1 hr.AC → 50% cold working → 700°C. X 1 hr.AC	975
	1100°C. X 1 hr.solution treatment → hot working → 700°C. X 1 hr.AC → 50% cold working → 700°C. X 1 hr.AC → 50% cold working → 700°C. X 1 hr.AC	1000
	*1100°C. X 1 hr. solution treatment → → hot working** → 700°C. X 1 hr.AC → 50% cold working → 700°C. X 1 hr.AC	925
Conventional Treatment	*1100°C. X 1 hr. solution treatment ⟶ hot working* → 900°C. X 1 hr.AC 50% cold working	875

"*" indicates mean cooling speed after hot working 8°C./min.
"**" indicates mean cooling speed after hot working 8°C./min.

Table 6 shows the austenitic grain-coarsening temperature of the steels F and G treated by the respective processes.

TABLE 6

Treating Processes and Austenitic Grain-Coarsening Temperatures		
(Types of Steels: F and G)		
Type of Steels	Process	Coarsening Temp. (°C.)
This Invention	*1100°C. solution treatment X 1 hr.→ hot working* → 700°C. X 1 hr.AC → 50% cold working → 700°C. X 1 hr.AC	1000
	1100°C. solution treatment X 1 hr. →hot working → 50%cold working →700°C. X 1 hr.AC	975
F	*1100°C. solution treatment X 1 hr. →hot working* → 900°C. X 1 hr.AC →50% cold working → 700°C. X 1 hr.AC	950
	1100°C. solution treatment X 1 hr. →hot working → 700°C. X 1 hr.AC →50% cold working →700°C. X 1 hr.AC →50% cold working →700°C. X 1 hr.AC	1000
Conventional	*1100°C. solution treatment X 1 hr. →hot working* →900°C. X 1 hr.AC→ 50% cold working	925
This Invention	*1100°C. X 1 hr. solution treatment → hot working* → 700°C. X 1 hr.AC→ 50% cold working→ 700°C. X 1 hr.AC	950
G		
Conventional	*1100°C. solution treatment X 1 hr. →hot working* → 900°C. X 1 hr.AC →50% cold working	875

"*" indicates mean cooling speed after hot working 8°C./min.

Having now fully described the invention, it will be apparent to one of ordinary skill in the art that many changes and modifications can be made thereto without departing from the spirit or scope of the invention. Accordingly,

What is claimed as new and intended to be covered by Letters Patent is:

1. A method of processing steel material which is characterized by an abnormal grain growth curve of austenitic grain after cold working and which has a high grain coarsening temperature which comprises:

heating said cold worked steel material to a temperature of between the ferrite recrystallization temperature and the A_1 transformation temperature for a period of at least 5 minutes so as to effect recrystallization of the ferritic structure which had become deformed during cold working, and thereby raising the austenitic grain coarsening temperature,

further heating said steel material to a temperature within the austenitic region of from the A_1 transformation temperature to the austenitic grain coarsening temperature to effect austenitizing to form a non-mixed grain structure.

2. A method of processing steel material which contains a grain refining element which is characterized by an abnormal grain growth curve of austenitic grain after cold working and which has a high grain coarsening temperature which comprises:

heating said cold worked steel to a temperature of between the ferrite recrystallization temperature and the A_1 transformation temperature for a period of at least 5 minutes so as to effect recrystallization of the ferritic structure which had become deformed during cold working, and thereby raising the austenitic grain coarsening temperature, and to precipitate said grain refining elements in the form of nitride, carbide and/or oxide,

further heating said steel material to a temperature within the austenitic region of from the A_1 transformation temperature to the austenitic grain coarsening temperature to effect austenitizing to form a non-mixed grain structure.

3. The method of claim **2,** wherein said steel material is hot worked subsequent to the first heating stop and prior to cooling in the 900° – 500°C range.

4. The method of claim **2,** wherein cold working is sequentially cycled with intermediate heat treating, between a first cold working operation and each succeeding cold working operation, said intermediate heat treatment being below the temperature of the A_1 transformation point.

5. The method of claim **3,** wherein cold working is sequentially cycled with intermediate heat treating, between a first cold working operation and each succeeding cold working operation, said intermediate heat treatment being below the temperature of the A_1 transformation point.

6. The method of claim **2,** wherein said steel is cold worked by machining.

7. The method of claim **3,** wherein said steel is cold worked by machining.

8. The method of claim **1,** wherein after recrystallization said steel material is subjected to carburization treatment in the austenitic region.

* * * * *