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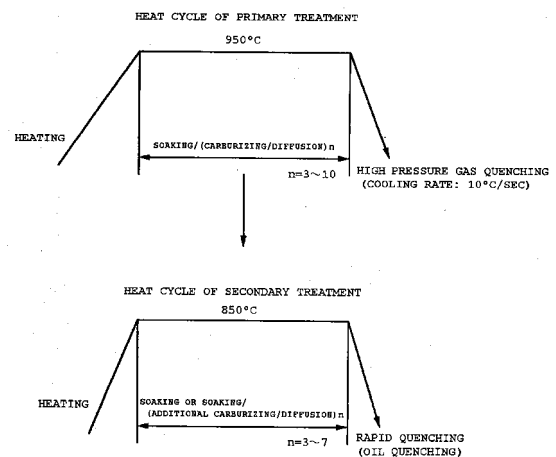
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(54) **HIGH-CONCENTRATION CARBURIZED/LOW-STRAIN QUENCHED MEMBER AND PROCESS FOR PRODUCING THE SAME**

(57) The present invention can provide a super carburized, low-distortion quenched member with both higher performance and minimized heat-treatment distortion attained in the field of super carburized quenching which features high strength, high contact pressure and the like. A process for the production of the super carburized low-distortion quenched member includes a primary treatment and a secondary treatment. The primary treatment comprises heating a steel member for a machine structure to a temperature within an austenite region by vacuum carburizing (low-pressure carburizing) to have carbon dissolved at least at a eutectoid carbon concentration of a surface layer portion of the member and then quenching the member at a cooling rate of from 3 to 15°C/sec from the temperature within the austenite region to a temperature not higher than an A₁ transformation point to have at least one of ultrafine carbide and nuclei of the carbide formed in the surface layer portion of the member. The secondary treatment comprises subsequently heating and soaking the member to a temperature within the austenite region and then conducting rapid quenching to have ultrafine carbide precipitated in a range of from 10 to 30% in terms of effective case depth percentage in an outermost surface layer portion.

FIG. 3



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Description**Technical Field**

5 **[0001]** This invention relates to carburizing and quenching treatment widely used as a reinforcement method for machine structural members, more specifically to a super carburized, quenched member featuring temper softening resistance, high strength, high contact pressure and the like, especially to a super carburized, low-distortion quenched member (which may hereinafter be referred to simply as "member") with mutually conflicting properties, that is, higher performance and heat-treatment distortion attained together and also to its production process.

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Prior Art

15 **[0002]** Owing to excellent properties such as high fatigue strength and wear resistance, carburized and quenched members (hereafter referred to as "case hardened members") are widely used as various members in transport equipment, industrial machines and the like. From the viewpoint of dimensional reductions, weight reductions and/or the like through further improvements in the performance of such members, numerous developments have been made on case hardened members. Recently, the vacuum carburizing (low-pressure carburizing) process has been developed. Compared with the conventional gas carburizing process, the vacuum carburizing process has excellent characteristic features such as environmental friendliness, the prevention of intergranular oxidation, the feasibility of high-temperature carburizing treatment, and easy control of carburizing and carbon diffusion, and therefore, is expected to find still broader utility from the standpoints of further improvements in the performance and quality of members and further improvements in their productivity.

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25 **[0003]** As a method for providing a machine structural member such as a gear or axle member with improved pitting resistance by applying carburizing and quenching to the member, there is carbonitriding treatment. According to this treatment, carbon and nitrogen are caused to concurrently diffuse into the matrix of a member such that the member can be provided with improved temper softening resistance. In addition, there has also been developed super carburizing treatment to have carbide precipitated in a surface layer portion of a member such that the member can be provided with improved temper softening resistance. Keeping in step with evolutions in low-pressure carburizing facilities, a great deal of research has been conducted in recent years.

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35 **[0004]** As a representative example of the super carburizing treatment, Patent Document 1 discloses a carburizing treatment process for a member. According to Patent Document 1, it is proposed to form quasispheroidal or spheroidal carbide at a volume percentage of 30% or higher within a range up to a depth of 0.4 mm by conducting precarburing to such a carbon content that spheroidal carbide is caused to precipitate in a surface layer portion of a steel member and the carbon concentration in the surface layer portion becomes not higher than A_{cm} but not lower than a eutectoid concentration between steel and carbon, slowly cooling or quenching the thus-treated member to convert the surface layer portion into a bainite, pearlite or martensite structure, and then heating the member at a ramp rate of not greater than $20^{\circ}\text{C}/\text{min}$ from the A_{c1} point to a temperature in a range of from 750 to 950°C to effect carburizing and quenching.

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45 **[0005]** According to the above-described process, the member can be improved in properties such as pitting properties owing to the precipitation of the carbide in the surface layer portion of the member. Nonetheless, the resulting member involves problems such as a deformation and distortion by heat treatment, because the process is super carburizing that causes the precipitation of the carbide as much as 30% in the surface layer portion.

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55 **[0006]** As a method for causing carbide to precipitate in an ultrafine form in a surface layer portion of a member by super carburizing, many heating and cooling methods have been investigated. In Patent Document 1, it is described to be desirable that subsequent to the precarburing, air cooling (which forms a bainite or pearlite structure) or quenching (which forms a martensite structure) is conducted, and that in the carbide-forming treatment as the next step, the member is heated at a slow ramp rate of not greater than $20^{\circ}\text{C}/\text{min}$ from the A_{c1} transformation temperature to a temperature within the range of from 750 to 950°C , and after direct quenching or air cooling, the member is again heated and quenched.

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[0007] Further, Patent Document 2 and Patent Document 3 propose, as an optimal method, to conduct slow cooling (or $30^{\circ}\text{C}/\text{hr}$ or less) after precarburing or primary carburizing.

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[0008] When the quenching after the precarburing or primary carburizing is conducted by air cooling or slow cooling in the method disclosed in Patent Document 1, 2 or 3, however, a network of carbide tends to precipitate along grain boundaries in a surface layer portion of a member. The next step, that is, the carbide-forming treatment can hardly break up the network of carbide in a short time to have the carbide distributed and precipitated within the surface layer portion. To overcome this shortcoming, heating and subsequent cooling may be conducted a plurality of times in some instances.

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[0009] On the other hand, Patent Document 1 also discloses quenching with an aim directed toward forming a martensite structure by increasing the cooling rate of a member subsequent to its precarburing. This technique, however, involves a potential problem that carbide nuclei in a surface layer portion may dissolve out. It is also concerned that the quenching may take place with supersaturated carbon, and due to high-carbon martensitic transformation, the member

may develop a greater deformation or distortion through an expansion, shrinkage or the like.

[0010] Patent Document 4 discloses a production process of a case hardened member by low-pressure carburizing. There is a reference to the conversion of carbide into an ultrafine form such as the control of the carbon concentration at 0.5 to 0.7 wt.% in primary carburizing and at 0.7 to 1 wt. % in secondary carburizing and the control of primary cooling at a very slow rate of from 1 to 10°C/min. Concerning deformation strain, however, this production process is not expected to be preferred like the above-mentioned Patent Documents 1, 2 and 3.

[0011] Just for readers' information, a description is now made of some advantages of low-pressure carburizing, which is finding wide-spread commercial utility in recent years, over conventional gas carburizing.

- a) A change from a carburizing step to a diffusion step can be readily and promptly modified.
- b) High-temperature treatment is feasible so that prompt carburizing can be conducted.
- c) No intergranular oxidation takes place in a surface layer portion of a member, and in the member under treatment, it is hence possible to inhibit the occurrence of cracks which would otherwise begin to take place from such a defect.
- d) No sooting takes place, thereby causing no uneven carburizing which would otherwise take place as a result of sooting.

Patent Document 1: JP-B-62-24499

Patent Document 2: JP-B-2787455

Patent Document 3: JP-B-2808621

Patent Document 4: JP-A-2002-348615

Disclosure of the Invention

Problem to be Solved by the Invention

[0012] Even in super carburizing by the conventional low-pressure carburizing, however, no optimal balance can be achieved between the progress of formation of carbide within a surface layer portion of a member under treatment and the microstructure of the surface layer portion. The problem of a deformation or strain of the treated member, therefore, still remains unresolved. As a consequence, grinding, strain-correcting finishing or the like is essential for the member after the carburizing step. Such additional work has led to a reduction in the inherent ability of super carburizing that permits use under higher contact pressure, a reduction in productivity and an increase in manufacturing cost, thereby preventing the popularization of super carburizing treatment.

Means for Resolving the Problem

[0013] The present invention has resolved the above-described problem by developing an optimal process, which makes it possible to use a member under a higher contact pressure and also to provide the member with a lower strain while making use of low-pressure carburizing facilities that permit a variety of control promptly with higher accuracy as to the concentration of carbon in the member, the repetition of carburizing treatment/diffusion treatment, and diverse temperature conditions, heating conditions and cooling rate (quenching) conditions for heating, soaking, carburizing, quenching and the like of the member.

[0014] The above-described problem can be resolved by the present invention as defined below:

1. A process for producing a super carburized, low-distortion quenched member, which comprises a primary treatment of heating a steel member for a machine structure to a temperature within an austenite region by vacuum carburizing (low-pressure carburizing) to have carbon dissolved at least at a eutectoid carbon concentration of a surface layer portion of the member and then quenching the member at a cooling rate of from 3 to 15°C/sec from the temperature within the austenite region to a temperature not higher than an A₁ transformation point to have at least one of ultrafine carbide and nuclei of the carbide formed in the surface layer portion of the member; and a secondary treatment of subsequently heating and soaking the member to a temperature within the austenite region and then conducting rapid quenching to have ultrafine carbide precipitated in a range of from 10 to 30% in terms of effective hardened depth percentage in an outermost surface layer portion.

[0015] 2. A production process as described above, wherein in the secondary treatment, additional carburizing treatment is applied to the surface layer portion of the member.

3. A production process as described above, wherein in the secondary treatment, the ultrafine carbide is caused to precipitate in the surface layer portion of the member to form a structure composed primarily of martensite and

containing a mixed structure of troostite and retained austenite or the like in parts thereof such that the outermost layer portion (a portion A) of the layer, a layer portion (a portion B) inner than the portion A and a layer portion (a portion C) inner than the portion B are in an order of $A \geq C \geq B$ in terms of the fineness of austenite grain size.

5 [0016] A super carburized, low-distortion quenched member comprising a surface layer portion of a structure composed primarily of martensite and containing a mixed structure of troostite and retained austenite or the like in parts thereof, wherein in the surface layer, an outermost surface layer (a portion A), a layer (a portion B) inner than the portion A and a layer (a portion C) inner than the portion B are in an order of $A \geq C \geq B$ in terms of the fineness of austenite grain size.

10 **Advantageous Effects of the Present Invention**

[0017] The process according to the present invention performs the treatment of a member in low-pressure carburizing facilities while making the combined use of the primary treatment of conducting adequate super carburizing and quenching at an optimal cooling rate and the secondary treatment of subsequently causing a fine carbide to simply and efficiently precipitate; and can minimize the deformation and strain of the member treated through the heat treatment. Owing to the adoption of this process, the greatest concern about the conventional super carburizing, for example, the cumbersome grinding, strain correction and the like of the member after the treatment, such as the bending of an axle or the deformation strain of a tooth profile, can be substantially relieved, thereby bringing about advantageous effects that significant improvements can be made in the productivity, quality and cost of the case hardened member.

20 [0018] According to the process of the present invention, additional carburizing treatment may be applied to the surface layer portion of the member in the secondary treatment. This additional carburizing treatment makes it possible to achieve a high hardness of matrix and also to reduce the crystal grain size of an outermost surface layer portion of the member to an ultrafine grain size and, therefore, is also extremely effective for providing the member with higher strength and higher toughness. By the process of the present invention, it is possible to readily achieve higher strength, higher toughness, higher contact pressure and the like for members such as axles and gears to which super carburizing has heretofore been hardly applicable. Therefore, the process according to the present invention can be widely applied to fields where there is a high need for such properties, and has an advantageous effect that it can make significant contributions to improvements in the performance of a member and also to reductions in the size and weight of the member.

30 **Best Modes for Carrying out the Invention**

[0019] Based on best modes for carrying out the invention, the present invention will next be described in further detail. The followings are the course of technical endeavors and the findings, which have led to the present invention. With a view to developing a super carburizing process for causing ultrafine carbide to precipitate in a surface layer portion of a member by using low-pressure carburizing facilities, the present inventors carried out a thorough investigation on possible relations between the concentration of carbon in the surface layer portion and various heating and cooling conditions and the precipitation form of the ultrafine carbide in the surface layer portion and the microstructure of the matrix. Concerning improvements or the like in strain by heat treatment while assuming members such as gears and axles, research and development was also conducted from many directions. An aim was then set at the establishment of a novel process for super carburizing and low-strain quenching, which can achieve both of mutually-conflicting properties of providing a member with higher performance by super carburizing and minimizing a deformation, distortion or the like of the member while balancing them at high levels.

40 [0020] Upon applying super carburizing to a surface layer portion of steel (member), the most important point is to have ultrafine carbide precipitated as much as possible in a surface layer portion of the member through the optimal combination of the primary treatment and the secondary treatment. In the control of the formation of the ultrafine carbide, carburizing and quenching facilities also play an important role. In the present invention, a variety of developments were conducted while using low-pressure carburizing facilities that compared with conventional carburizing facilities, permit a variety of control promptly with higher accuracy as to the concentration of carbon in the member, the repetition of carburizing treatment/diffusion treatment, and diverse temperature conditions, heating conditions and cooling rate (quenching) conditions for heating, soaking, carburizing, quenching and the like of the member.

50 [0021] Described specifically, a variety of investigations were conducted on the heating, soaking, super carburizing, diffusion and cooling (quenching) conditions of a member during the primary treatment to firstly reduce the deformation or strain of the member at the stage of the primary treatment. In the secondary treatment as the next step, carburizing and quenching (cooling) conditions are important to permit adjustments or the like in the precipitation of ultrafine carbide and the grain size of austenite in the carburized layer. Specifically, it has been found that in the secondary treatment, the deformation or strain of a member by the heat treatment can be minimized by controlling a range, in which the ultrafine carbide precipitate in a surface layer portion of the member, to 10 to 30% in terms of effective case depth percentage and further by converting an outermost surface layer portion into an ultrafine crystalline structure.

[0022] The term "effective case depth percentage" as used herein means a ratio (t/T) of a precipitated depth (t) of ultrafine carbide existing in an outermost surface layer portion of a member to an effective case depth (T) of the member after completion of the secondary treatment (including the tempering treatment at 180°C). It is to be noted that the term "effective case depth" means a distance from a surface of a hardened layer, which is still in a quenched state or has been tempered at a temperature not exceeding 200°C, to the position of a critical depth of a Vickers hardness (HV) of 550 as measured by the Method of Measuring Case Depth Hardened by Carburizing Treatment for Steel (JIS G0557). Next, the term "precipitated depth of ultrafine carbide" means the maximum depth, where the ultrafine carbide exists, from the outermost surface layer portion of the member as determined by an analysis under an optical microscope or an electron microscope. To facilitate the discrimination of the ultrafine carbide, the member is analyzed in a state of being etched with an etching solution such as 5% nital etching reagent.

[0023] The vacuum carburizing (low-pressure carburizing) facilities for use in the present invention are equipped with a carburizing and heating chamber including a treatment furnace which is sectionally controllable at different pressures of from 200 to 2,000 Pa, and are available on the market. Conventionally-available vacuum carburizing facilities are all usable in the present invention. As the primary treatment in the present invention, the member is heated and soaked to a predetermined temperature in the furnace of the facilities, and to raise the concentration of carbon in the surface layer portion of the member to or higher than the eutectoid carbon concentration, the member is then quenched at an appropriate cooling rate. In the subsequent secondary treatment, the carbide is caused to precipitate in an ultrafine form in the surface layer portion of the member, optionally followed by additional carburizing treatment as needed.

[0024] According to the primary treatment in the process of the present invention, steel to be treated (member) is heated and soaked to an austenite region of from 900 to 1,100°C, carburizing is conducted such that the carbon concentration of a surface layer portion becomes preferably 0.8 wt.% or higher, and from the thus-carburized state, quenching is then conducted at an optimal cooling rate. Optimal cooling conditions are to evenly cool the member at a cooling rate of from 3 to 15°C/sec over a temperature range of from the carburizing temperature (the temperature in the austenite region) to the A_1 transformation temperature or lower, preferably to 400°C or lower. By this cooling, ultrafine carbide is caused to precipitate in the surface layer portion of the member so that a structure composed primarily of martensite is formed in the surface layer portion. The term "ultrafine carbide" means an $M_{23}C_6$ type carbide formed as a result of bonding of carbide-forming elements such as Cr and Mo in Fe_3C (cementite) or steel with carbon dissolved in super-saturation.

[0025] In the secondary treatment, the non-carburized portion (interior) of the member is heated and soaked to a range of from an austenizing temperature to the austenizing temperature +80°C, preferably to a range of from 10 to 70°C above the austenizing temperature, and is then rapidly quenched to effect precipitation of ultrafine carbide such that the carbon concentration of the surface layer portion becomes preferably 0.8 wt.% or higher, more preferably 1.0 to 2.0 wt.%. It is preferred to apply, in parallel with the precipitation of the ultrafine carbide in the surface layer portion, additional carburizing treatment to the surface layer portion to promote the precipitation of the ultrafine carbide in the surface layer portion, and from the state that the carbon concentration of the matrix has been adequately adjusted, to further conduct rapid quenching.

[0026] The temperature of the final quenching after the secondary treatment varies depending on the pretreatment conditions, that is, whether the final quenching is after the heating and soaking or after the heating, soaking and additional carburizing. The rapid quenching can be conducted at the temperature after the pretreatment or at a temperature raised or lowered relative to the temperature of the pretreatment. In other words, the temperature of the final quenching after the secondary treatment can be set at a level commensurate with the quality of heat treatment such as the hardness and microstructure required for the member.

[0027] With a view to establishing optimal conditions for super carburizing, the present inventors conducted a detailed investigation on the carbon concentrations upon heating, soaking and carburizing and diffusion and various cooling (quenching) conditions with respect to the primary treatment in which super carburizing is applied to a surface layer portion of a member in low-pressure carburizing facilities and the secondary treatment in which ultrafine grains of carbide are caused to precipitated in the surface layer portion. As a result, it was succeeded in obtaining a super carburized, quenched member having a carbon concentration of preferably 0.8 wt.% or higher, more preferably from 1.0 to 2.0 wt.% in a range of from 10 to 30% in terms of the percentage of an effective case depth (t/T) in an outermost surface layer portion and having a three-layer structure consisting of a superultrafine grain layer of No. 10 or greater austenite grain size, a fine grain layer and an ultrafine grain layer in this order from the outermost surface layer. It has been found that the super carburized, quenched member is minimized in deformation or distortion after the treatment and that the correction of a strain, which has been unavoidable in the conventional super carburizing, can be obviated or can be readily conducted compared with the conventional process.

Examples

[0028] Based on certain Examples, the present invention will next be described in further detail.

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Machine structural steels (materials) shown in Table 1 were provided. Those materials were subjected beforehand to normalizing treatment at 900°C and were then machined to prepare stepped round-bar test pieces of $\phi 30/\phi 25/\phi 20 \times L300$ mm, respectively. As carburizing and quenching of each test piece, the primary treatment of the super carburizing step in the present invention was conducted using facilities which permitted heating and carburizing at a low pressure and also permitted oil hardening and high pressure gas cooling.

[0029] It is to be noted that steel grades 1 and 2 are carburizing, quenching steels as specified under the JIS, steel grade 1 is SCM420, chromium-molybdenum steel, and steel grade 2 is SCr415, chromium steel. MAC14 as steel grade 3 is a grade for a commercial product developed by a steel maker, and is steel developed by increasing the Cr content in comparison with the above-described two steel grades and further adding Mo element with a view to causing $M_{23}C_6$ type ultrafine carbide to precipitate upon super carburizing (the primary and secondary treatments).

[0030]

Table 1: Used Steels and Their Chemical Components

		(wt. %)						
	Steel grade	C	Si	Mn	P	S	Cr	Mo
1	SCM420	0.20	0.30	0.75	0.019	0.025	1.10	0.20
2	SCr415	0.16	0.35	0.78	0.021	0.019	1.05	0.02
3	MAC14	0.15	0.27	0.53	0.020	0.022	2.50	0.38

[0031] Table 2 summarizes the results obtained by experimenting in various ways effects of the cooling rate on the states of carbide to be precipitated in surface layer portions of test pieces and the deformations of the test pieces by heat treatment through the primary treatment in the present invention. As conditions for the primary treatment, super carburizing of each test piece was conducted by the heat cycle shown in FIG. 1 such that subsequent to heating and soaking, an effective case depth of 0.5 mm would be achieved. Described specifically, super carburizing and diffusion treatment of each test piece were alternately conducted at 950°C for about 70 minutes, respectively, such that the carbon concentration of the surface layer portion of the test piece in its final state would be controlled at about 1.5wt.%. From a state that the carbon concentration of the surface layer portion of each test piece was in supersaturation, quenching of the test piece was conducted under the corresponding cooling rate condition shown in Table 2 to investigate the shape and size of the carbide in the surface layer portion of the test piece and the microstructure of the surface layer portion of the test piece.

[0032] To determine the deformations and strains of the above-described steel grades by the primary treatment, stepped round-bar test pieces ($\phi 30/\phi 25/\phi 20 \times L300$ mm) of the respective steel grades were provided as test pieces. In a state of being supported at opposite ends, each test piece was analyzed for a runout at its axial central part to investigate a relationship between the cooling rate and the axial of the test piece.

[0033]

Table 2: Relationships between Cooling Conditions for Primary Treatment and Precipitation Form of Carbide and Runout

Ex./Comp.Ex.	No.	Steel grade	Cooling rate (°C/sec)	Shape and size of carbide	Microstructure of surface layer portion	Runout-TIR (mm)
Comp. Ex.	1	SCM420	1	Flaky, 3-10 μm	F + P + B	0.45
Ex.	2	Same as above	12	Granular, 0.5-5 μm	M + T	0.17
Comp.Ex.	3	Same as above	20	Granular, <2 μm	M + γ	0.38
Comp.Ex.	4	SCr415	1	Flaky, 3-10 μm	F + P	0.40
Ex.	5	Same as above	4	Granular, 0.5-5 μm	M + T	0.15

(continued)

Ex./Comp.Ex.	No.	Steel grade	Cooling rate (°C/sec)	Shape and size of carbide	Microstructure of surface layer portion	Runout-TIR (mm)
Comp.Ex.	6	MAC14	1	Granular + flaky, 5 μm	F + P + B	0.38
Ex.	7	Same as above	7	Flaky, 2-7 μm	M + T	0.20

TIR: Total Indicating Reading

[0034] The signs shown in the table and analysis methods of the properties shown there will now be described below.

- 1) The cooling rate indicates an average cooling rate at the axial central part of each test piece from the quenching temperature of 950°C after the completion of the carburizing and diffusion for the test piece to 400°C.
- 2) The shape and size of carbide was observed under a scanning electron microscope.
- 3) Abbreviations for microstructures F: ferrite, P: pearlite, B: bainite, T: troostite, M: martensite, γ: retained austenite.
- 4) The radial runout indicates a runout of a test piece, which was mounted on a both-end supporting, runout measuring instrument, as measured at a central part of the test piece by a dial gauge.

[0035] In each of the comparative examples shown as Test Piece Nos. 1, 4 and 6 in Table 2, the cooling rate during the cooling was as low as 1°C/sec so that the carbide precipitated in the surface layer portion consisted primarily of a network of carbide formed of carbide flakes bonded together and the matrix was in the form of a slack quenching structure of ferrite, pearlite and bainite. As a consequence, those comparative examples were all large in radial runout and deformation. The comparative example shown as Test Piece No. 3, on the other hand, was subjected to rapid cooling equivalent to conventional oil quenching (20°C/sec). Its surface layer portion contained a very small amount of precipitated carbide, and had a structure quenched from a high carbon state that carbon was in supersaturation. That comparative example was large in radial runout and deformation.

[0036] When the cooling rate was 4 to 12°C/sec as in each of the examples as Test Piece Nos. 2, 5 and 7 (the present invention), ultrafine carbide precipitated in a large amount, and moreover, microstructures appeared as nuclei for the ultrafine carbide, leading to improvements in the deformation and distortion (runout) of the test piece as the outstanding serious problems of super carburizing. Described specifically, compared with slow cooling that cooling is slow or rapid quenching that cooling is fast in contrast, the radial runout of each of the test pieces according to the present invention was of approximately a half level of the radial runouts in the rest of the examples, thereby realizing a substantial reduction in radial runout. From these results, the cooling rate during the quenching in the primary treatment is optimally 3 to 15°C/sec.

[0037] Table 3 shows the results obtained by using representative ones of the test pieces subjected to the primary treatment shown in Table 2, applying the secondary treatment in various ways to the representative test pieces to cause ultrafine carbide to finally precipitate in their surface layer portions, and investigating the carbon concentrations, states of precipitated carbide, microstructures, crystal grain sizes, etc. in their surface layer portions and the radial runouts of the test pieces. As conditions for the secondary treatment, the heat cycle shown in FIG. 2 was followed, the soaking temperature was selectively set at three levels of 800°C, 850°C and 900°C, all above the A_1 transformation temperature, and subsequent to the heating and soaking, additional carburizing was also conducted at the same time to achieve a carbon concentration higher than the eutectoid carbon concentration as a technique for further raising the carbon concentrations in the surface layer portions and also increasing the amounts of precipitated ultrafine carbide through the secondary treatment.

The subscript "n" in (carburizing/diffusion)_n or (additional carburizing/diffusion)_n in FIGS. 1 through 3 means the number of repetitions of carburizing or diffusion in the corresponding step, and is set in commensurate with the quality required for each member. In the case of Test Piece No. 2 shown as an example in Table 2, for example, n was set at 8 (n = 8), and in the case of Test Piece No. 2-2 shown as an example in Table 3, on the other hand, n was set at 5 (n = 5).

[0038]

Table 3: Relationships between Treatment Conditions for Secondary Treatment and Precipitation Form of Carbide and Runout

Ex. / Comp.Ex.	No.	Steel grade	Tempera- ture of secondary treatment (°C)	Additional carburizing	Precipitation of carbide		Austenite grain size and three-layer structure	
					Shape	Amount	Outermost surface layer	Three layers
Comp.Ex.	2-1	SCM420	900	Applied	Ultrafine	A little	≥10	Included
Ex.	2-2	Same as above	850	Applied	Ultrafine	Adequate	≥10	Included
Comp.Ex.	2-3	Same as above	800	Applied	Flaky	Excessive	≥10	Included
Ex.	5-1	SCr415	850	Applied	Ultrafine	Adequate	≥10	Included
Ex.	5-2	Same as above	850	Not applied	Ultrafine	A little	6-8	Not included
Ex.	7-1	MAC14	850	Applied	Ultrafine	Adequate	≥10	Included
Ex.	7-2	Same as above	850	Not applied	Ultrafine	A little	6-8	Not included

Table 3 (Cont'd)

Ex. / Comp.Ex.	No.	Steel grade	Micro- structure	Carbon concent- ration (%)	Effective case depth (mm)	Percentage of effective case depth, t/T (%)	Runout·TIR (mm)
Comp.Ex.	2-1	SCM420	M + γ	1.6	0.52	5	0.35
Ex.	2-2	Same as above	M	1.5	0.48	25	0.14
Comp.Ex.	2-3	Same as above	M + F	1.5	0.46	20	0.20
Ex.	5-1	SCr415	M	1.6	0.50	18	0.16
Ex.	5-2	Same as above	M	1.2	0.46	10	0.23
Ex.	7-1	MAC14	M	1.4	0.53	25	0.21
Ex.	7-2	Same as above	M + γ	1.1	0.48	15	0.25

TIR: Total Indicating Reading

[Analysis method of carbon concentration surface layer portion]

[0039] Using each of the test pieces ($\phi 30/\phi 25/\phi 20 \times L300$ mm), chips were collected by lathe turning from the surface layer portion to the 0.05 mm depth of its $\phi 25$ mm portion, and the carbon concentration of the surface layer portion was determined by a chemical analysis.

[0040] From Table 3, the Test Piece No. 2 series indicate effects on the precipitation form of carbide and others when the secondary treatment temperature was varied, and the Test Pieces No. 5 and No. 7 series indicate effects on the precipitation of ultrafine carbide and the final carbon concentrations in the surface layer portions depending on whether or not the additional carburizing was applied in the secondary treatment.

[0041] Concerning the secondary treatment temperature (which may hereinafter be called "the additional carburizing temperature"), the temperature of 900°C employed for Test Piece No. 2-1 involves a problem in that the carbide in a surface layer portion dissolves to lead to a reduction in the overall precipitation of carbide grains and also to an increase

in the radial runout of the test piece. With the secondary treatment temperature of 800°C employed for Test Piece No. 2-3, carbide flakes precipitate at grain boundaries in the surface layer portion, and the core portion of the member is quenched incomplete. Test pieces, therefore, develop variations in radial runout. From these results, the optimal temperature for the treatment that causes ultrafine carbide to precipitate in a surface layer portion by the secondary treatment can preferably be a temperature equivalent to the A_3 transformation temperature + 10-70°C, which is determined by the composition of the member (before the carburizing treatment).

[0042] As to whether or not the additional carburizing treatment is applied in the secondary treatment, the application of the additional carburizing treatment has been recognized, as evident from the results of Test Piece Nos. 5-1 and 7-1, to bring about the advantageous effect that carbide precipitates in an ultrafine form, to say nothing of an improvement in the concentration of carbon in the surface layer portion. As a reason for the advantageous effect, it may be contemplated that, as the carbon in the surface layer portion precipitate as carbide and the concentration of carbon in the matrix becomes lean, the replenishment of carbon to the surface layer portion by the additional carburizing could promote the new formation of ultrafine carbide, such as Fe_3C and $M_{23}C_6$, and nuclei thereof.

[0043] As shown in FIG. 4, it has also been found that in the member subjected to the additional carburizing treatment, the austenite grain size of the outermost surface layer portion is reduced to an ultrafine grain size. The term "ultrafine grain size" corresponds to an austenite grain size of No. 10 or greater as measured by the carburized grain-size testing method in JIS-G0551, "Method of Testing Austenite Grain Size for Steel". A significant characteristic feature has also been discovered in that a three-layer structure formed of fine grains and ultrafine grains is formed extending toward the inside. Paying attention to a relationship between the austenite grain size and the carburized layer, the grain sizes of the outermost surface layer portion greatest in the amount of precipitated ultrafine carbide, the carburized layer portion (fine grain portion) located inside the outermost surface layer portion and the ultrafine grain portion located still inside the fine grain portion are in a relationship of $A \geq C \geq B$, in which "A", "C" and "B" stand for the outermost surface layer portion, the ultrafine grain portion and the fine grain portion, respectively. Incidentally, the austenite grain size of a surface layer portion in conventional carburizing is generally equivalent to No. 7 or 8. In the present invention, the surface layer portion has a grain structure of the characteristic three-layer structure which does not appear in the conventional carburizing treatment.

[0044] As an advantageous effect of such an ultrafine grain layer, it has a significant characteristic feature in that the toughness of a hardened surface layer, said toughness having been a concern about conventional carburized members, can be improved and high toughness can also be imparted to the carburized layer itself in addition to the feasibility of higher contact pressure as a characteristic feature of the present invention, and therefore, is extremely effective for providing carburized members with still higher strength from now on.

[0045] Table 4 shows effects of the percentage of an effective case depth of a carbide layer precipitated in super carburizing according to the present invention on various properties. Various test pieces were prepared by providing SCM420, JIS steel for machine structure, as a material, subjecting the material to normalizing treatment at 900°C beforehand, and then machining the resultant material. The super carburizing of each test piece was conducted by the heat cycle of primary treatment and secondary treatment shown in FIG. 3. Each treated test piece was analyzed and investigated for pitting life, impact strength, distortion by heat treatment, etc. Concerning effects of the carbon concentration of the outermost surface layer portion of each test piece shown in Table 5, the test piece was treated by the heat cycle shown in FIG. 3 in a similar manner as the various test pieces in Table 4, and the carbon concentration and the like of the treated test piece were investigated.

[0046] The adjustment of the precipitation depth of carbide in Table 4 was effected primarily by the control or the like of the carburizing time and carbon concentration, and the adjustment of the carbon concentration of the outermost surface layer portion in Table 5 was effected by controlling the process gas flow, treatment time and the like upon repeating carburizing and diffusion in the primary treatment and secondary treatment in accordance with a program calculated beforehand. Process gases for low-pressure carburizing include propane, acetylene, ethylene and the like. Among these, most popular and economical propane was used. As an inert gas upon diffusion, on the other hand, nitrogen gas was used. Further, the rapid quenching in the secondary treatment was conducted by oil. As an alternative, the rapid quenching can also be conducted by high pressure gas which makes sole or mixed use of gases such as N_2 , He and H_2 .

[0047]

Table 4: Effects of the Percentage of Effective Case Depth on Strength, Durability and Distortion by Heat Treatment

Ex./ Comp.Ex.	Sign	Percentage of effective case depth, t/T (%)	Carburizing time (min)	Carbon concentration of the outermost surface layer portion (%)	Rolling fatigue life (number of rotations)	Impact strength (J)	Roundness (μ)
Comp.Ex.	A	5	80	1.0	6.5×10^6	118	29
Ex.	B	10	104	1.5	1.1×10^7	110	31
Ex.	C	20	119	1.7	2.1×10^7	105	39
Ex.	D	30	134	1.9	2.3×10^7	98	50
Comp.Ex.	E	40	149	2.0	2.2×10^7	67	65

[0048]

Table 5: Effects of the Carbon Concentration of Outermost Surface Layer Portion on Strength, Durability and Distortion by Heat Treatment

Ex./ Ref.Ex.	Sign	Carbon concentration of outermost surface layer portion (%)	Carburizing time (min)	Rolling fatigue life (number of rotations)	Impact strength (J)	Roundness (μ)	Additional carburizing
Ref.Ex	F	<0.8	80	5.3×10^6	56	30	Not applied
Ex.	G	1.0	80	1.5×10^7	87	32	Same as above
Ex.	H	1.5	80	2.0×10^7	69	37	Same as above
Ex.	I	1.0	96	1.9×10^7	116	35	Applied
Ex.	J	1.5	133	2.4×10^7	111	39	Same as above
Ex.	K	2.0	130	2.6×10^7	98	53	Same as above

[0049]

1) The percentage of effective case depth indicates the ratio (t/T) of the depth (t) of an ultrafine carbide layer to a case depth (T) of 550 HMV or greater in terms of micro-Vickers hardness.

2) The rolling fatigue life indicates the number of repetitions of rotation until occurrence of pitting under the below-described conditions. Contact pressure: 3 GPa, rotation speed: 1,500 rpm, slipping ratio: -40%, oil temperature: 80°C.

3) The impact strength indicates destructive energy as measured using a Charpy test piece.

4) The roundness indicates the amount of a deformation of the inner diameter of a ring in the X-Y direction as measured by a profile measuring instrument while using as the ring a test piece in a ring form of $\phi 100(\phi 80) \times 15t$.

[0050] A description will now be made about effects of the percentage of effective case depth on the rolling fatigue life. When an ultrafine carbide layer was as shallow as 5% in terms of the percentage of effective case depth as in the comparative example represented by the sign A, it is considered that the amount of precipitated ultrafine carbide itself was small and therefore, that the treated test piece did not have temper softening resistance, which is characteristic to super carburizing, and was low in pitting toughness. In the case of the comparative example represented by the sign E in which the percentage of effective case depth was 40%, the high hardness range was broadened, resulting in a problem that the impact strength was reduced, and with respect to a deformation by heat treatment as determined in terms of roundness, there was also a tendency toward increased distortion. From these results, the percentage of effective

hardened depth in a precipitated carbide layer is optimally in a range of from 10 to 30%.

[0051] A description will next be made about effects of the carbon concentration of the outermost surface layer portion shown in Table 5 on the pitting life. It is considered that the signs H, J and K, in each of which the carbon concentration of the outermost surface layer portion was high, were superior in pitting life and that in the cases of the signs G and I in each of which the carbon concentration was 1%, that is, lower compared with the former signs, they were somewhat inferior in pitting life. When the carbon concentration of the outermost surface layer portion is lower than 0.8 wt.% as in the sign F shown as a referential example, the test piece was significantly inferior in pitting toughness. Namely, the greater the amount of ultrafine carbide precipitated in the outermost surface layer portion and the higher the carbon concentration of the outermost surface layer portion, the better the pitting life. Accordingly, the carbon concentration of super carburizing can be set preferably at 0.8 wt.% or higher in the present invention.

[0052] Regarding the upper limit to the carbon concentration through carburizing, no particular problem arose up to 2.0 wt.%. An increase in carbon concentration to a still higher level in excess of 2.0 wt.% involves a potential concern that precipitation of carbide flakes may be facilitated and the impact strength and deformation by heat treatment of the test piece may tend to become disadvantageous. It is, therefore, necessary to set the carbon concentration of the outermost surface layer portion at a level commensurate with properties required for the member (test piece).

[0053] A description will next be made about effects of the additional carburizing treatment in the secondary treatment in the signs I, J and K on the pitting life, impact strength and deformation (strain) by heat treatment. Compared with the signs G and H in each of which the carbon concentrations was similar but the additional carburizing was not applied, the signs I, J and K varied less in all the properties and were better. As a reason for this advantage, it can be contemplated that the additional carburizing treatment may stabilize the carbon concentration of the matrix and may also promote the formation of ultrafine carbide in the outermost surface layer portion, the carburized layer itself may be converted into a dense and well-balanced structure, and the quality available through the heat treatment may be thoroughly stabilized.

[0054] From the above-described various analysis results, it is desired, as optimal treatment conditions in the process of the present invention, to employ machine structural steel as a member, to conduct super carburizing as a combination of the primary treatment and the secondary treatment in low-pressure carburizing facilities to treat the member under optimal heating and cooling conditions, and then to control the final step such that the depth of precipitated carbide falls within the range of from 10 to 30% in terms of the percentage of effective case depth and the carbon concentration of the surface layer becomes 0.8 wt.% or higher.

Industrial Applicability

[0055] As appreciated from the above-described series of results, the present invention can provide an absolutely novel, super carburized, low-distortion quenched member and its production process. According to the present invention, machine structural members such as gears and axle members can be provided with higher strength and can be used under higher contact pressure, thereby making it possible to materialize with low distortion the needs for various members of higher strength, higher performance, lighter weight and smaller size, such as members required to have low distortion, rotary sliding or reciprocal sliding members equipped with bearing structures, and members required to have high contact fatigue resistance and high abrasion resistance under high contact pressure.

Brief Description of the Drawings

[0056]

[FIG. 1] Heat cycle of the primary-treatment.

[FIG. 2] Heat cycle of the secondary treatment.

[FIG. 3] Heat cycle of the examples.

[FIG. 4] Optical micrograph (magnification: $\times 100$) of Test Piece No. 2-2 in Table 3.

Claims

1. A process for producing a super carburized, low-distortion quenched member, which comprises a primary treatment of heating a steel member for a machine structure to a temperature within an austenite region by vacuum carburizing (low-pressure carburizing) to have carbon dissolved at least at a eutectoid carbon concentration of a surface layer portion of said member and then quenching said member at a cooling rate of from 3 to 15°C/sec from said temperature within said austenite region to a temperature not higher than an A_1 transformation point to have at least one of ultrafine carbide and nuclei of said carbide formed in said surface layer portion of the said member; and a secondary treatment of subsequently heating and soaking said member to a temperature within said austenite region and then

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conducting rapid quenching to have ultrafine carbide precipitated in a range of from 10 to 30% in terms of effective case depth percentage in an outermost surface layer portion.

- 5
2. A process according to claim 1, wherein in said secondary treatment, additional carburizing treatment is applied to said surface layer portion of said member.
- 10
3. A process according to claim 2, wherein in said secondary treatment, said ultrafine carbide is caused to precipitate in said surface layer portion of said member to form a structure composed primarily of martensite and containing a mixed structure of troostite, retained austenite and the like in parts thereof such that said outermost layer portion (a portion A) of said layer, a layer portion (a portion B) inner than said portion A and a layer portion (a portion C) inner than said portion B are in an order of $A \geq C \geq B$ in terms of the fineness of austenite grain size.
- 15
4. A super carburized, low-distortion quenched member comprising a surface layer portion of a structure composed primarily of martensite and containing a mixed structure of troostite and retained austenite or the like in parts thereof, wherein in said surface layer, an outermost surface layer (a portion A), a layer (a portion B) inner than said portion A and a layer (a portion C) inner than said portion B are in an order of $A \geq C \geq B$ in terms of the fineness of austenite grain size.
- 20
- 25
- 30
- 35
- 40
- 45
- 50
- 55

FIG. 1

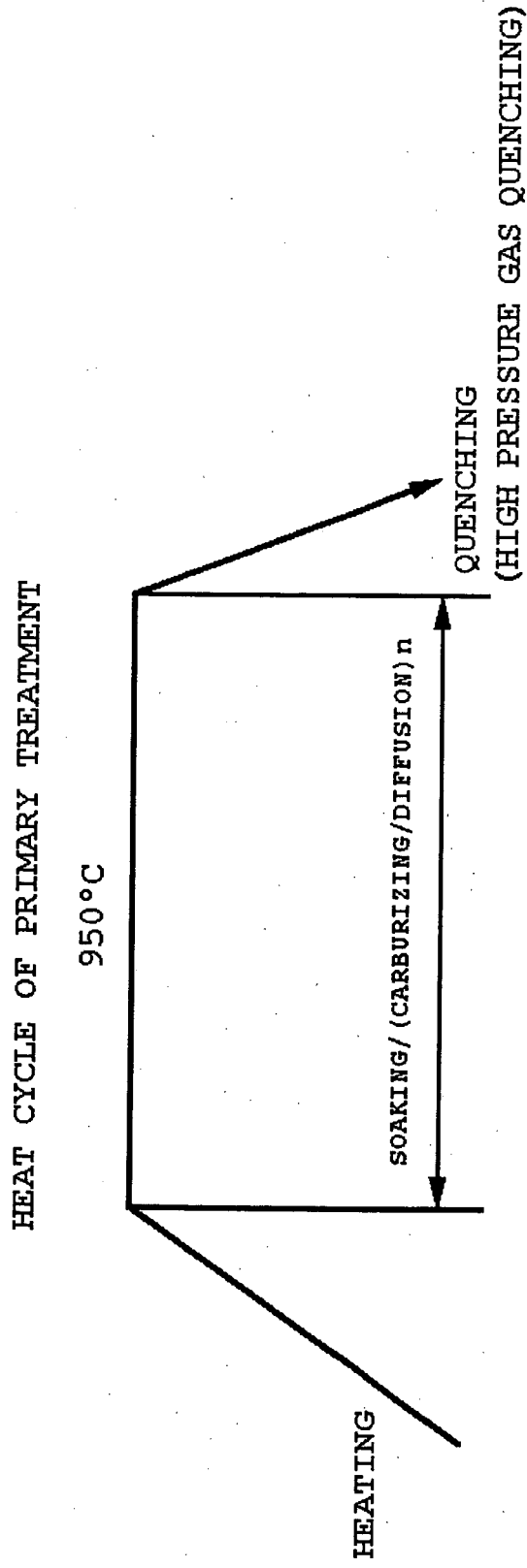


FIG. 2

HEAT CYCLE OF SECONDARY TREATMENT

800°C/850°C/900°C

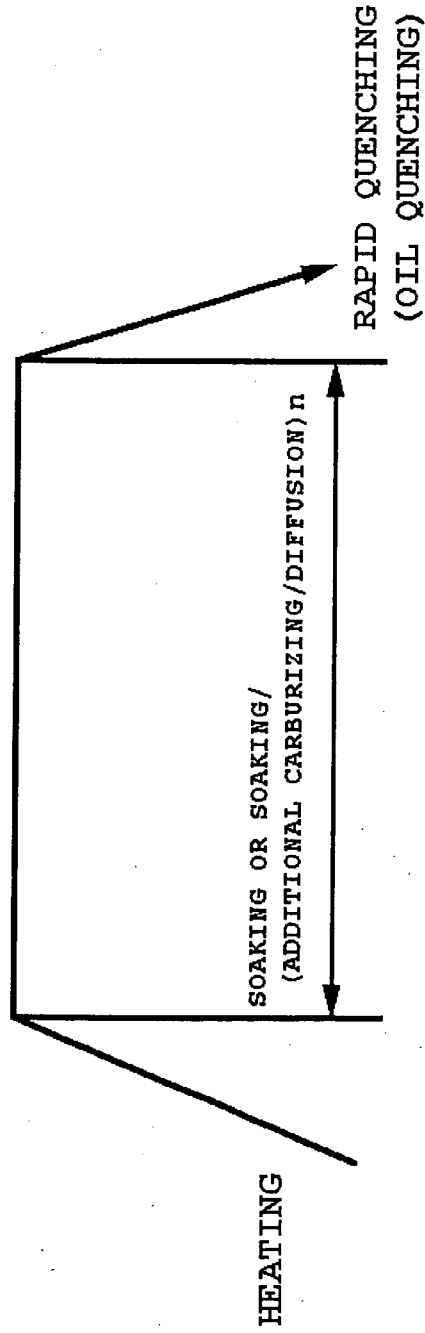


FIG. 3

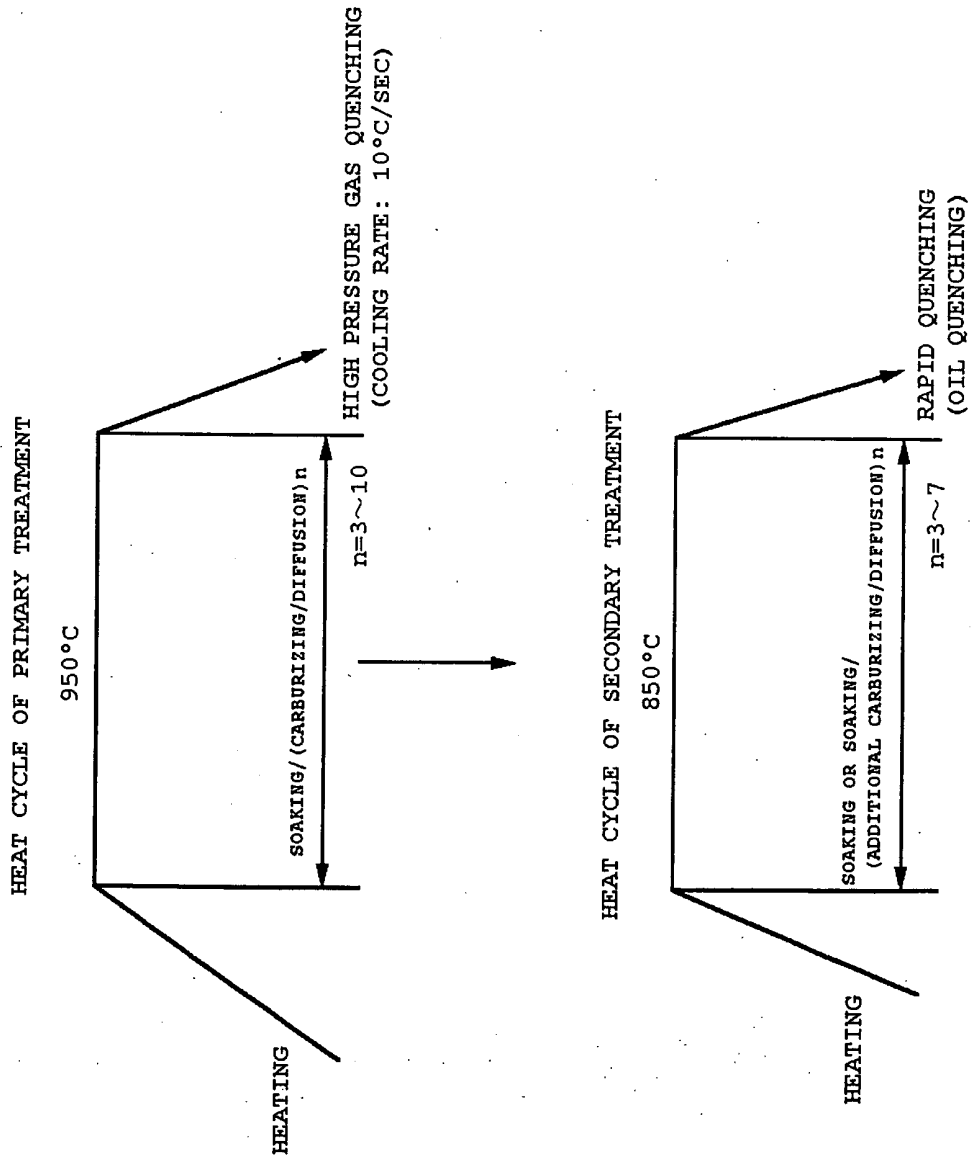
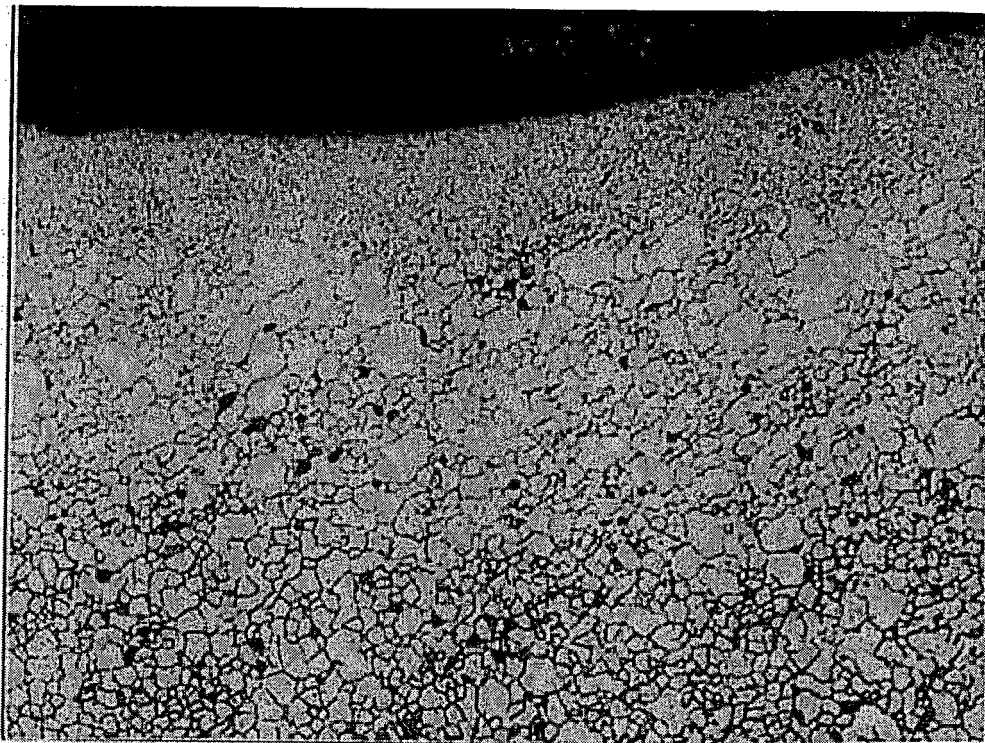


FIG. 4



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2006/302161

A. CLASSIFICATION OF SUBJECT MATTER <i>C23C8/22</i> (2006.01), <i>C23C8/80</i> (2006.01), <i>C21D1/06</i> (2006.01), <i>C21D9/00</i> (2006.01), <i>C22C38/00</i> (2006.01), <i>C22C38/22</i> (2006.01)		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED		
Minimum documentation searched (classification system followed by classification symbols) C21D1/06, C21D9/00, C22C38/00, C22C38/22, C23C8/22, C23C8/80		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2006 Kokai Jitsuyo Shinan Koho 1971-2006 Toroku Jitsuyo Shinan Koho 1994-2006		
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	JP 8-003720 A (Sumitomo Metal Industries, Ltd.), 09 January, 1996 (09.01.96), Claim 2; Par. Nos. [0007], [0078] (Family: none)	1-4
A	JP 8-081737 A (Daido Steel Co., Ltd.), 26 March, 1996 (26.03.96), Par. Nos. [0004], [0047]; Fig. 2 (Family: none)	1-4
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/> See patent family annex.		
* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention	
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Date of the actual completion of the international search 06 March, 2006 (06.03.06)	Date of mailing of the international search report 14 March, 2006 (14.03.06)	
Name and mailing address of the ISA/ Japanese Patent Office	Authorized officer	
Facsimile No.	Telephone No.	

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2006/302161

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2002-212642 A (NTN Corp.), 31 July, 2002 (31.07.02), Claims 1 to 3; Par. Nos. [0001], [0008], [0018]; Fig. 13 (Family: none)	1-4

REFERENCES CITED IN THE DESCRIPTION

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