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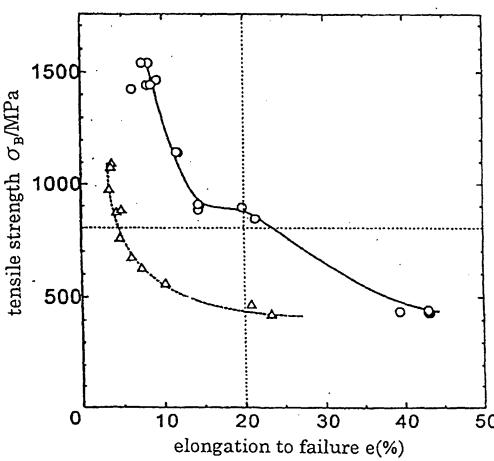
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(54) HIGH STRENGTH AND HIGH DUCTILITY STEEL PLATE HAVING HYPERFINE CRYSTAL GRAIN STRUCTURE PRODUCED BY SUBJECTING ORDINARY LOW CARBON STEEL TO LOW STRAIN WORKING AND ANNEALING, AND METHOD FOR PRODUCTION THEREOF

(57) A high strength and high ductility low carbon steel having a tensile strength of 800MPa or more, an uniform elongation of 5% or more, and an elongation to failure of 20% or more which is produced by a method comprising subjecting an ordinary low carbon steel or an ordinary low carbon steel added with boron in an amount being 0.01% or less and effective for acceler-  
ing martensitic transformation to processing and heat

treatment to prepare a product having coarser size of austenite crystal grains and then to water-quenching, to provide a steel product having a martensite phase in an amount of 90% or more, and subjecting the steel product to a low strain processing, specifically a cold rolling at a total rolling reduction in thickness of 20% or more and less than 80%, and to a low temperature annealing at 500°C to 600°C, and a method for producing said high strength and high ductility low carbon steel.

Fig. 7



**Description****FIELD OF THE INVENTION**

**[0001]** The present invention relates to a high strength and high ductility low carbon steel having a tensile strength of 800MPa or more, an uniform elongation of 5% or more, and an elongation to failure of 20% or more which is produced by a method comprising (1) subjecting an ordinary low carbon steel or an ordinary low carbon steel added with boron in an amount of 0.01% or less being effective for accelerating martensitic transformation to processing and heat treatment to prepare a steel sheet having coarser austenite crystal grains and then to water-quenching, to provide a steel sheet having a martensite phase in an amount of 90% or more, and (2) subjecting said steel sheet to a low strain cold-rolling of a total rolling reduction of thickness 20% or more and less than 80%, and to a low temperature annealing at 500°C to 600°C, and a method for producing said high strength and high ductility low carbon steel.

**[0002]** In the present invention, the ordinary low carbon steel means a steel whose carbon content is 0.2% or less, manganese content is 1.6% or less, silicon content is 0.5% or less, phosphorus content is 0.05% or less and sulfur content is 0.05% or less. The ordinary low carbon steel added with minute amount (0.01% or less) of boron means the steel produced by adding effective amount of boron necessary for acceleration of martensitic transformation in an amount of 0.01% or less to above mentioned ordinary low carbon steel for the purpose to improve the quenching property.

**[0003]** In the present invention, content % means weight %.

**BACKGROUND OF THE INVENTION**

**[0004]** In recent years, the improvement in usability of vacant space accompanying the high-rise building, energy saving requirement for cars or ships and recycling of natural resources are becoming more requisite, and these tendency is also applicable to the steel materials. To satisfy former two requirements, it is necessary to make the strength and ductility of the steel sheet much higher, and in order to improve in recycling of natural resources as well, it is necessary to achieve said improvement in making the strength and ductility of the steel sheet much higher by using ordinary low carbon steel not by adding other alloying elements.

**[0005]** In order to develop the steel sheet of high degree properties required several project teams are established. These project teams are named as, for example, Super Metal Project or Super Steel Project and are aiming to develop a ferrite structure steel having "800MPa" tensile strength, which is two times to ordinary low carbon steel, having high ductility, and having property for easy welding as well, by producing ultra fine crystal grains of 1 μm or less in the present "400MPa

class composition steel sheet".

**[0006]** In the concerned technical field, for the improvement in strength by refining the ferrite crystal grains of steel, it is well known that the relationship of Hall-Petch equation is realized, that is, yield stress and tensile strength are improved by refining the size of ferrite crystalline of steel and simultaneously the toughness is also improved. However, there is a problem of the elongation falling down in tension test.

**[0007]** In CAMP-ISIJ Vol.11 (1998), pp 1031-1034, the following disclosure is reported. In studing to obtain a steel whose strength is improved to 800 MPa grade of 400 MPa grade steel with good weldability as a starting material, they settled their object of their study as to accomplish the grain size of 1μm or less in ferrite-carbide structure. And, as the concrete measures to accomplish said object, following process is mentioned. The austenite transforming treatment is carried out on a specimen having 8 mm thickness, namely, after said specimen is heat treated at the temperature of 1000°C for 60sec, cooled down by water so as to obtain martensite structure, then the biaxial hot rolling is carried out on the specimen at a total rolling reduction of thickness 90% at 640°C. And they reported that the ferrite structure of the obtained steel is characterized to have an equiaxed fine structure, the nominal grain size becomes 0.77μm and Vickers hardness is 245, which is corresponding to tensile strength of 760 MPa. However, in said reference, there is no description reporting the actual measuring procedure about the tensile strength by preparing a test piece for strength test from the obtained bulk steel, further, there is no mention concerning elongation. Still more, the steel used in said reference is the steel whose manganese content is increased to 2.03% for the purpose to obtain the quenching ability, further the rolling of the martensite structure is carried out by hot condition at 640°C.

**[0008]** Further, in the development of a steel which satisfies the requirement, such as high strength, high toughness and high ductility, the solid-solution hardening method which adds alloy element, the precipitation hardening method and the transformation strengthening method are being investigated, however, these methods have a problem of high price because of containing high amount of alloy element, further have a problem to deteriorate the recycling property. On the other hand, to solve said problems, the strengthening methods by refining of crystalline grains, which are the methods by adding no alloy element, are investigated and reported, however, since these methods are based on a large strain processing, the problem of requiring a particular processing equipment arises.

**[0009]** The inventors of the present invention have already investigated about the structure and the mechanical properties of a steel sheet obtained by the combination of Accumulative Roll-Bonding (called as ARB) at room temperature and annealing, which is a large strain processing, using the steel sheet whose structure is fer-

rite-pearlite as a starting material. However, since the structure obtained after large strain processing has a heterogeneous structure in which both a region containing cementite and a region not containing cementite exist, a heterogeneous mixed grains structure whose grain size of ferrite are not uniform is generated in annealing process, therefore, the expected high strength and high ductility steel sheet could not be obtained.

**[0010]** The idea of producing the ultra fine ferrite crystalline grains structure of ordinary low carbon steel from a martensite structure is not a novel one, because said idea is also used by STX-21 Project or Super Metal Project which promotes the development of super steel. However, by said method, the development to accomplish the high strength and high ductility low carbon steel having a tensile strength of 800MPa or more, an uniform elongation of 5% or more, and an elongation to failure of 20% or more has not realized yet. In particular, the idea to obtain a steel having high strength, high ductility and high toughness is not existing in the concept of these Projects.

**[0011]** The object of the present invention is to provide the steel sheet having said desired properties and a method to produce a steel sheet having said desired properties without big change of the producing plants for a conventional steel sheet.

**[0012]** As mentioned above, the idea to use a steel sheet with martensite structure as a starting material to realize the ultra fine ferrite crystal grain structure is a well known technique. However, it was considered to be difficult to form martensite structure overall in the ordinary low carbon steel whose quenching property is not so good in the process of producing said ordinary low carbon steel sheet.

**[0013]** In order to produce high strength and high ductility low carbon steel having a tensile strength of 800MPa or more, an uniform elongation of 5% or more, and an elongation to failure of 20% or more from a martensite steel as a starting material, as the first step, inventors of the present invention have studied the relationship between martensite steel as a starting material and the properties such as strength or ductility of low carbon steel obtained by a subsequent treatment. And on said studying we have found out that said high strength and high ductility low carbon steel having the expected strength, elongation and elongation to failure can be obtained from a steel whose martensite phase is 90% or more obtained by making the austenite crystalline grains coarser, and then quenching into water followed by a cold rolling at a total rolling reduction in thickness of 20% or more and less than 80% and by annealing, thus we have accomplished the object of the present invention.

**[0014]** Namely, the object of the present invention is accomplished by the combination of said low strain processing and annealing and the specific steel to be provided to said low strain processing and annealing.

## DISCLOSURE OF THE INVENTION

**[0015]** The 1<sup>st</sup> one of the present invention is a high strength and high ductility low carbon steel sheet having a tensile strength of 800MPa or more and an uniform elongation of 5% or more, which is produced by a method comprising, carrying out a low strain processing and annealing on a steel having a martensite phase in an amount of 90% or more obtained by coarsening the size of an austenite crystal grain, which is existing in an ordinary low carbon steel or an ordinary low carbon steel added with boron in an amount of 0.01% or less being effective for accelerating martensitic transformation, to 100μm or more and then quenching into water. Desirably, the 1<sup>st</sup> one of the present invention is the high strength and high ductility low carbon steel, wherein said steel possesses an ultra fine crystal grain ferrite structure having an average grain diameter of 1.0μm or less formed by a low temperature processing and annealing by carrying out a cold rolling at a total rolling reduction of thickness of 20% or more and less than reduction of thickness of 80%, and a low temperature annealing at the temperature range between 500°C or more and less than 600°C.

**[0016]** The 2<sup>nd</sup> one of the present invention is the method for producing a high strength and high ductility low carbon steel having a tensile strength of 800MPa or more and an uniform elongation of 5% or more comprising, carrying out a low strain processing and annealing on a steel sheet having a martensite phase in an amount of 90% or more obtained by coarsening the size of an austenite crystal grain, which is existing in an ordinary low carbon steel or an ordinary low carbon steel added with boron in an amount of 0.01% or less being effective for accelerating martensitic transformation, to 100μm or more and quenching into water, then carrying out a cold rolling at a total rolling reduction in thickness of 20% or more and less than 80%, and a low temperature annealing at the temperature range between 500°C or more and less than 600°C, to thereby form an ultra fine crystalline grain ferrite structure having an average grain diameter of 1.0μm or less.

## BRIEF ILLUSTRATION OF THE DRAWINGS

**[0017]**

Fig.1 is the optical microscopic (OM) picture showing the structure of the longitudinal-vertical cross sectional view of the ordinary low carbon steel plate (JIS-SS400, 2mm thickness) which is austenitized at 1000°C for 15 minutes, then quenching into water.

In the picture, RD indicates the rolling direction and ND indicates normal direction of the sheet.

Fig.2 is the optical microscopic picture showing the structure of the longitudinal-vertical cross sectional view of the cold rolled ordinary low carbon steel

(JIS-SS400) whose starting structure is a martensite structure. (a) shows the case of 50% cold rolling and (b) shows the case of 70% cold rolling.

Fig.3 shows the nominal-stress-nominal-strain curves of the quenched steel of the ordinary low carbon steel (JIS-SS400) and cold rolled steel of various rolling reduction in thickness. a is a cold rolled steel at a rolling reduction in thickness of 70%, b is a cold rolled steel at a rolling reduction in thickness of 50%, c is a cold rolled steel at a rolling reduction of thickness of 25%, d is a quenched steel of martensite structure, e is a steel as received of ferrite-pearlite structure

Fig.4 shows the nominal-stress-nominal-strain curves. a is a cold rolled steel at a rolling reduction in thickness of 50% of an ordinary low carbon steel (JIS-SS400) whose starting structure is martensite structure, and 30 minutes annealed steels of it (b; annealed at 400°C, c; annealed at 500°C, d; annealed at 550°C, e; annealed at 600°C).

Fig.5 shows the relationship between annealing temperature and mechanical properties of a cold rolled and annealed steel at a rolling reduction in thickness of 50% of an ordinary low carbon steel (JIS-SS400) whose starting structure is martensite structure.

- is tensile strength ( $\sigma_B$ ), -○- is 0.2% proof stress ( $\sigma_{0.2}$ ), -▲- is elongation of failure ( $\epsilon$ ), -△- is uniform elongation ( $\sigma_U$ ).

Fig.6 is the transmission electron microscopic (TEM) picture showing the structure of the longitudinal-vertical cross sectional view of a cold rolled and annealed steel at a rolling reduction of 50% of an ordinary low carbon steel (JIS-SS400) at various annealing temperatures whose starting structure is martensite structure.

Annealed at the temperature of (a) 400°C, (b) 500°C, (c) 550°C, (d) 600°C for 30 minutes.

Fig.7 is the graph showing the comparison of the relationship between tensile strength and elongation to failure (strength-ductility balance) of rolled and annealed steel at a rolling reduction of 50% of an ordinary low carbon steel (JIS-SS400) whose starting structure is martensite structure at the various annealing temperatures for 30 minutes (○), and that of rolled and annealed steel at a rolling reduction with ARB of 97% of the steel whose starting structure is ferrite-pearlite and annealed at various temperatures for 30 minutes (Δ).

Fig.8 is a JIS 5 test piece for elongation test.

## THE BEST EMBODIMENT TO CARRY OUT THE INVENTION

**[0018]** The present invention will be illustrated more in detail.

A. For the illustration of the present invention, the

method for test and apparatuses for measurement are illustrated.

5 1. The shape of a test piece used for the tensile test is 1/5 size of JIS 5 test piece (Fig.8) (gage length 10mm × gage width 5mm).

10 2. The specimen for optical microscopic (Nikon Co., Ltd., Opti Photo 100S) and TEM (Hitachi Co., Ltd., H-800) observation is prepared by a well-known method.

B. The important points of the present invention are illustrated with reference to the drawing.

15 The present invention will be illustrated along with following more concrete examples, however, following examples are mentioned only for easy understanding of the present invention and not intending to limit the scope of the present invention.

20 [0019] Fig.1 is an optical microscopic picture showing the structure of the longitudinal-vertical cross sectional view of a quenched steel which is obtained by using a hot rolled plate having 2mm thickness of the rolled steel material for a general construction use, namely, the

25 steel material containing minor constituents (JIS-SS400) such as C; 0.13%, Si; 0.01%, Mn; 0.37%, P; 0.02%, S; 0.004%, sol. Al; 0.04% as the receiving steel, and austenitization is carried out on said steel at 1000°C for 15 minutes so as to make coarse the size of

30 an austenite crystal grain to 100-200μm size, then water-quenched. This picture shows that the structure is the structure of coarse martensite structure containing about 4% of proeutectoid ferrite.

35 [0020] Fig.2 is an optical microscopic picture showing the structure of the longitudinal-vertical cross sectional view of a cold rolled steel obtained by cold rolling of the receiving steel of Fig.1 by multi pass cold rolling by a total rolling reduction in thickness of 50% (a) and 70% (b). The proeutectoid ferrite precipitated in prior austenite grains can be observed in black contrast. In general, it is said that the workability of martensite of carbon steel is not so good, however, from Fig.2 it is clearly understood that the low carbon steel martensite, at least the low carbon steel martensite prepared according to the

40 recipe of the present invention is possible to be cold rolled by reduction of 70% or more.

45 [0021] Fig.3 shows the nominal-stress-nominal-strain curves by tensile test of quenched steel of Fig.1 and cold rolled steel of Fig.2. For the reference, the nominal-stress-nominal-strain curve of a steel as received having ferrite-pearlite structure is shown by a dotted line. The tensile strength is improved from 410MPa to 1100MPa by quenching (d), further improved to 1340MPa by cold rolling of 25% (c), to 1470MPa by cold

50 rolling of 50% (b) and to 1640MPa by cold rolling of 70% (a). While, elongation to failure is 10% around in the case of quenched steel and 6% around in the case of cold rolled steel. And the uniform elongation of the cold

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rolled steel is 1% or less.

**[0022]** Fig.4 shows the nominal-stress-nominal-strain curves by tensile test of a cold rolled steel obtained by rolling reduction of 50% of Fig.3 and the annealed steels of it treated at various temperatures for 30 minutes. Although the strength is deteriorated by annealing, the ductility recovers by annealing at 500°C or more, and at the temperature of 500°C-550°C, the strength does not deteriorate so much, while the elongation to failure and the uniform elongation are obviously increased. Accordingly, in annealed steel at 550°C (d), the ultra high strength-high ductility steel of 870MPa tensile strength, 710MPa 0.2% proof stress, 21% elongation to failure and 8% uniform elongation is obtained.

**[0023]** Fig.5 shows the relationship between annealing temperature and tensile strength (-●), 0.2% proof stress (-○), elongation to failure (-▲) and uniform elongation (-△) of cold rolled steel by 50% and the annealed steel thereof. When annealing temperature exceeds 525°C, elongation to failure and uniform elongation are suddenly recovered, while tensile strength is almost fixed at the temperature between the range from 500°C to 550°C. This is the reason why the ultra high strength · high ductility steel is obtained.

**[0024]** Fig.6 is the TEM picture showing the structure of the longitudinal-vertical cross sectional view of a cold rolled and annealed steel at a rolling reduction of 50%. The picture indicates that the structure of 400°C annealed steel (a) is a lamella structure similar to a heavily rolled steel. In the case of 500°C annealed steel (b), ultra fine equiaxed grains of 100-300nm are observed in broad range. Not shown in the drawing, it already becomes clear from the limited range of vision electron diffraction pattern that these ultra fine equiaxed grains are surrounded by large angle grain boundaries and are not subgrains. The annealed steel at 550°C has also similar ultra fine equiaxed grain structure, however, at the annealing temperature of 600°C, the coarser grain whose grain size is grown to several  $\mu\text{m}$  and spherically precipitated cementite are observed.

**[0025]** It is understood that the precipitation of cementite occurs at the higher temperature than 500°C so as to restrict the growth of crystalline grain, and consequently the ultra fine crystalline grain structure of 100-300nm is generated, further the work hardening ability necessary for uniform elongation is provided simultaneously.

**[0026]** As mentioned above, by the use of the low carbon steel of martensite as a starting material, and by low strain processing of 50% rolling reduction and annealing at 550°C, ultra fine ferrite crystalline grain structure can be obtained, thus it becomes clear that it is possible to obtain a high strength and high ductility low carbon steel.

**[0027]** Fig.7 shows strength-ductility balance of 50% cold rolled and annealed steel of martensite which is a steel of the present invention (○) and large strain processed steel (97% cold rolling steel) whose starting structure is ferrite-pearlite structure of conventional art (△).

As mentioned above, when large strain processing is carried out using ferrite-pearlite structure as a starting structure, the structure obtained by annealing becomes mixed grain structure and desired high strength and high

5 ductility steel can not be obtained. On the contrary, in the case of cold rolled steel and annealed steel of martensite of the present invention, as clearly understood from Fig.7, the strength-ductility balance indicates experimental point which satisfies the conditions of 10 800MPa or more tensile strength and 20% or more elongation to failure is obtained.

## INDUSTRIAL APPLICABILITY

15 **[0028]** As mentioned above, in an ordinary low carbon steel of 0.13C (JIS-SS400), an ultra fine ferrite crystalline grain structure of 100-300nm grain size can be obtained by annealing after 50% cold rolling using martensite structure of the present invention as a starting structure, and by annealing at 550°C for 30 minutes, a steel which has excellent mechanical properties of 870MPa tensile strength, 21% elongation to failure and 8% uniform elongation is obtained. And it is obvious that the method for production of said steel provides excellent 20 effects, such as good economical advantage from the view point of facility and a satisfaction of social requirement from the view point of the environment and the circulation system of materials.

30 **Claims**

1. A high strength and high ductility low carbon steel sheet having a tensile strength of 800MPa or more, an uniform elongation of 5% or more and a elongation to failure of 20% or more, which is produced by a method comprising, carrying out a low strain processing and annealing on a steel product having a martensite phase in an amount of 90% or more obtained by coarsening the size of an austenite crystal grain, which is existing in an ordinary low carbon steel or an ordinary low carbon steel added with boron in an amount of 0.01% or less being effective for accelerating martensitic transformation, and then water-quenching.
2. The high strength and high ductility low carbon steel sheet of claim 1, wherein said steel possesses an ultra fine crystal grain ferrite structure having an average grain diameter of 1.0 $\mu\text{m}$  or less formed by carrying out a cold rolling at a total rolling reduction in thickness of 20% or more and less than 80%
3. The high strength and high ductility low carbon steel of claim 2, wherein the annealing process is carried out at the temperature range between 500°C or more and less than 600°C.

4. A method for producing a high strength and high ductility low carbon steel having a tensile strength of 800MPa or more and an uniform elongation of 5% or more and a elongation to failure of 20% or more comprising, carrying out a low strain processing and annealing on a steel sheet product having a martensite phase in an amount of 90% or more obtained by coarsening the size of an austenite crystal grain, which is existing in an ordinary low carbon steel or an ordinary low carbon steel added with boron in an amount of 0.01% or less being effective for accelerating martensitic transformation, to 100 $\mu$ m or more and water-quenching. 5

5. A method for producing a high strength and high ductility low carbon steel of claim 4, wherein the annealing is carried out at the temperature range between 500°C or more and less than 600°C after carrying out a cold rolling at a total rolling reduction in thickness of 20% or more and less than 80%. 15 20

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Fig.1

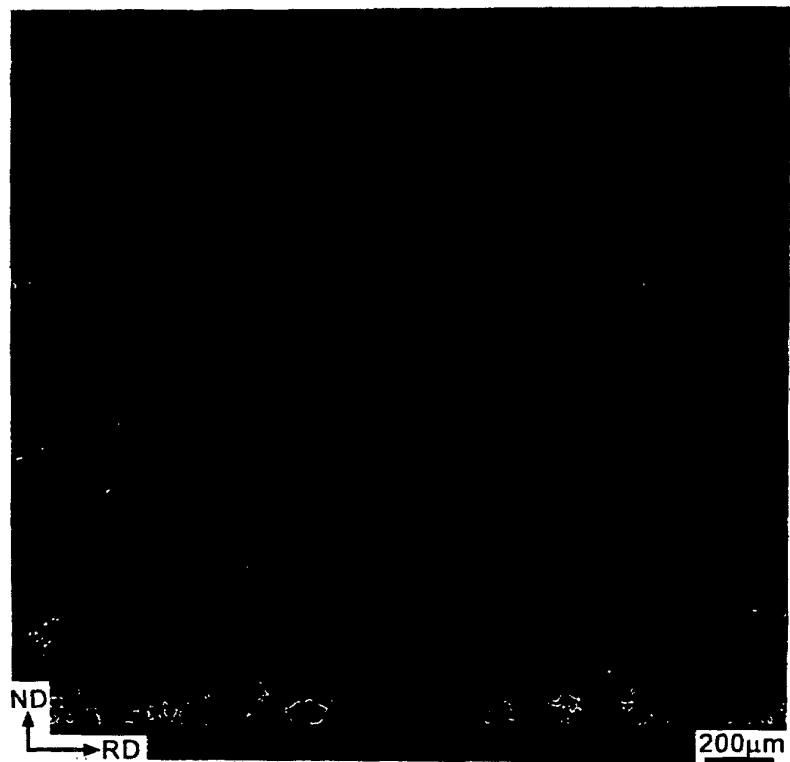


Fig.2

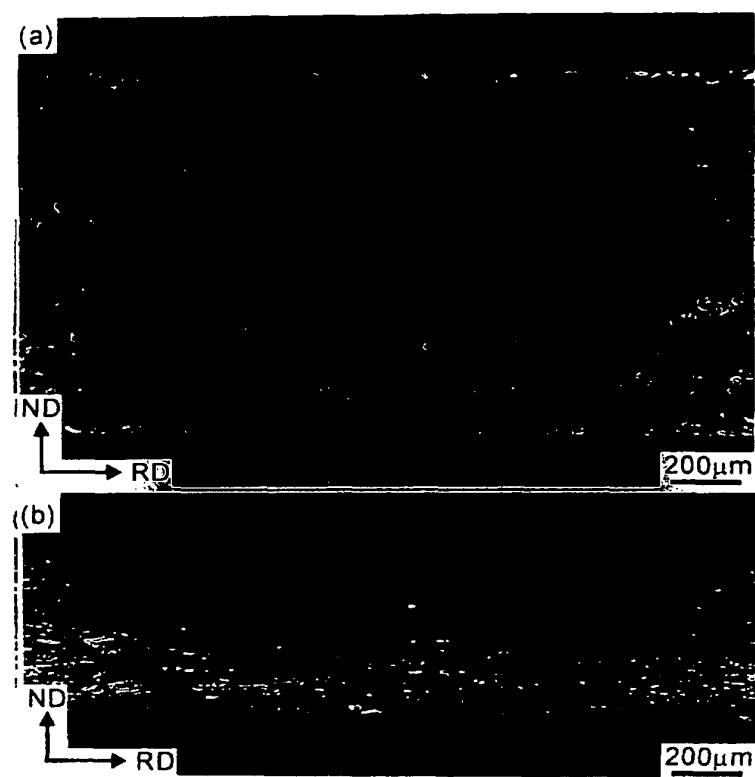


Fig.3

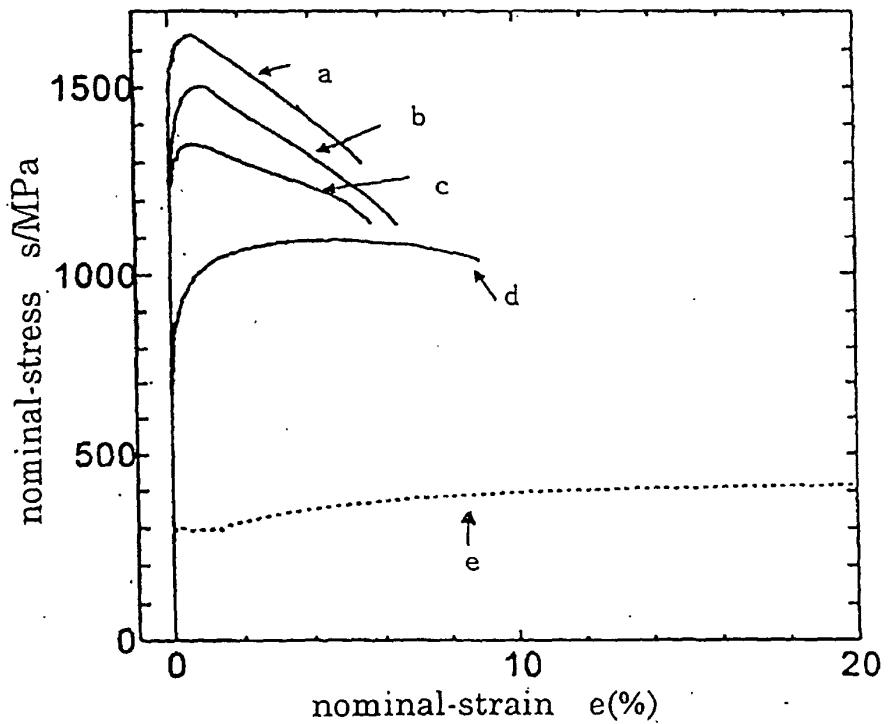


Fig.4

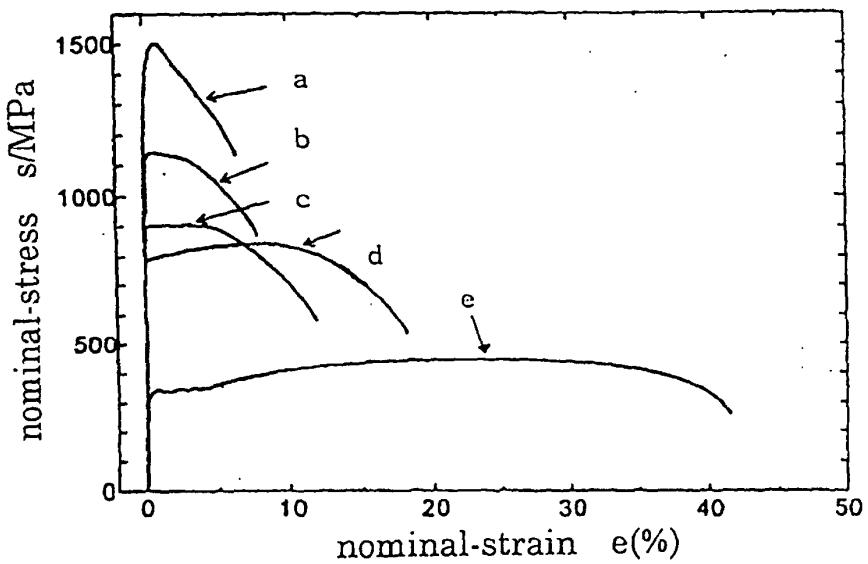


Fig.5

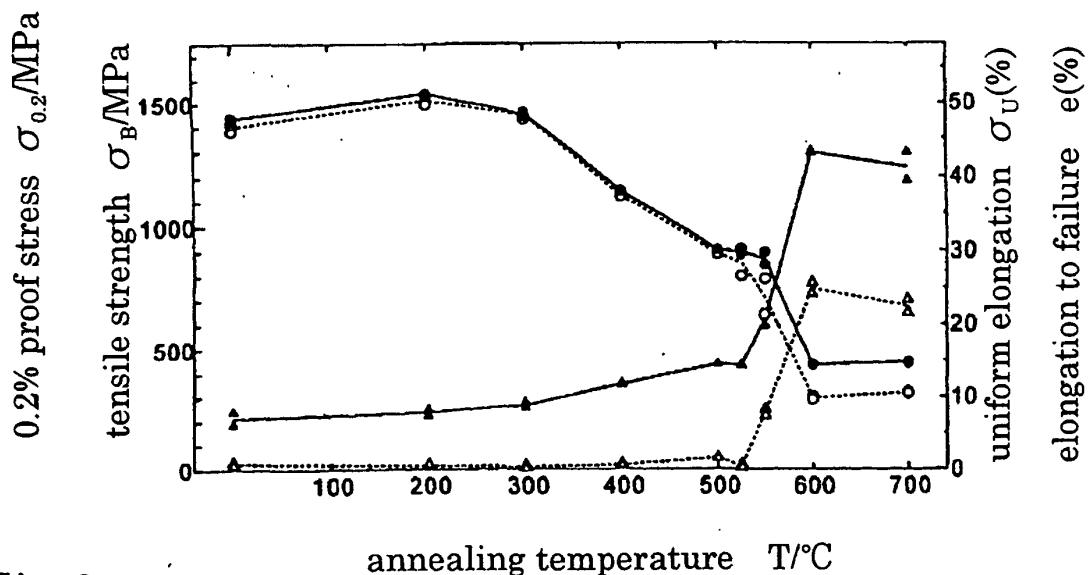


Fig.6

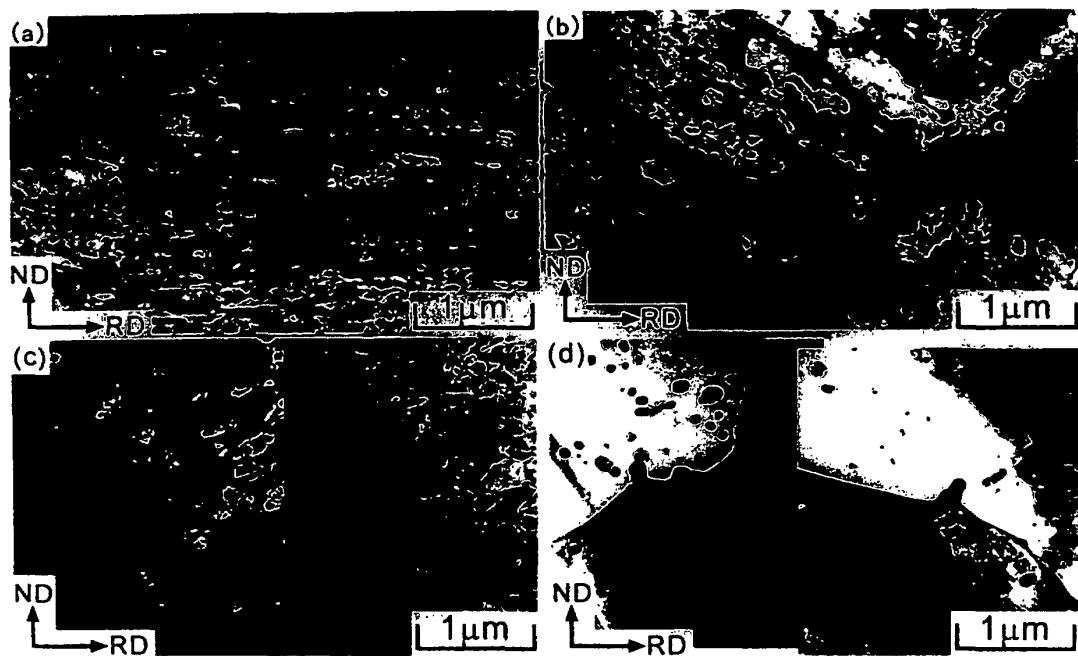


Fig.7

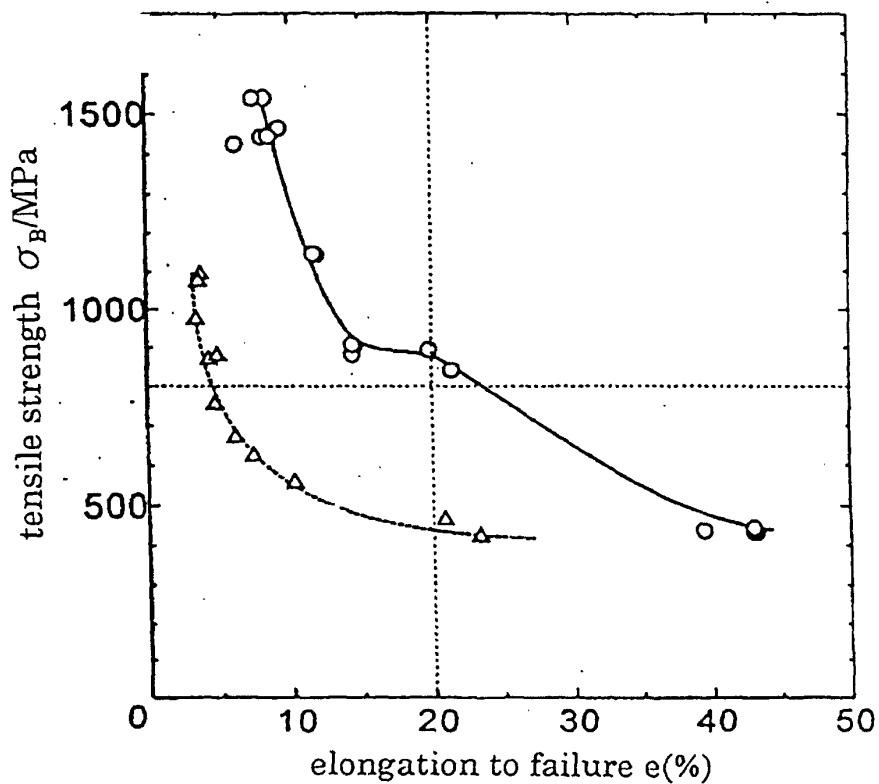
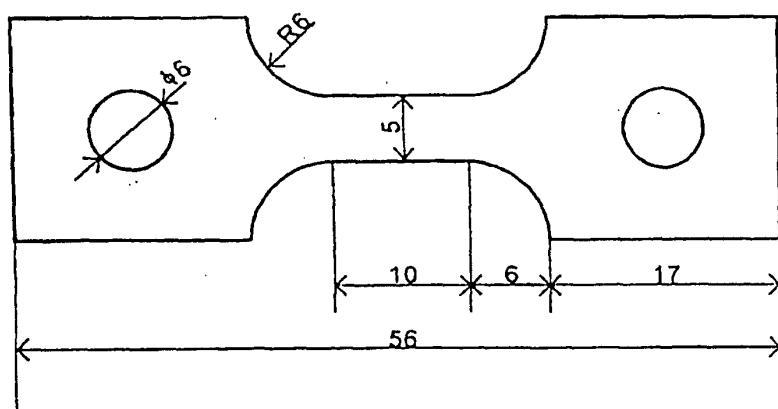


Fig.8



<b>INTERNATIONAL SEARCH REPORT</b>		International application No. PCT/JP02/02848									
<p><b>A. CLASSIFICATION OF SUBJECT MATTER</b> Int.Cl<sup>7</sup> C22C38/00, C21D8/00</p> <p>According to International Patent Classification (IPC) or to both national classification and IPC</p>											
<p><b>B. FIELDS SEARCHED</b> Minimum documentation searched (classification system followed by classification symbols) Int.Cl<sup>7</sup> C22C38/00, C21D8/00</p>											
<p>Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Jitsuyo Shinan Koho 1926-1996 Toroku Jitsuyo Shinan Koho 1994-2002 Kokai Jitsuyo Shinan Koho 1971-2002 Jitsuyo Shinan Toroku Koho 1996-2002</p>											
<p>Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) JOIS</p>											
<p><b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b></p> <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="text-align: left; padding: 2px;">Category*</th> <th style="text-align: left; padding: 2px;">Citation of document, with indication, where appropriate, of the relevant passages</th> <th style="text-align: left; padding: 2px;">Relevant to claim No.</th> </tr> </thead> <tbody> <tr> <td style="text-align: center; padding: 2px;">A</td> <td style="padding: 2px;">EP 0924312 A1 (Kawasaki Steel Corp.), 07 January, 1999 (07.01.99), Claims &amp; JP 11-80899 A</td> <td style="text-align: center; padding: 2px;">1-5</td> </tr> <tr> <td style="text-align: center; padding: 2px;">A</td> <td style="padding: 2px;">JP 2000-192139 A (Kawasaki Steel Corp.), 11 July, 2000 (11.07.00), Page 2 (Family: none)</td> <td style="text-align: center; padding: 2px;">1-5</td> </tr> </tbody> </table>			Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.	A	EP 0924312 A1 (Kawasaki Steel Corp.), 07 January, 1999 (07.01.99), Claims & JP 11-80899 A	1-5	A	JP 2000-192139 A (Kawasaki Steel Corp.), 11 July, 2000 (11.07.00), Page 2 (Family: none)	1-5
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.									
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A	JP 2000-192139 A (Kawasaki Steel Corp.), 11 July, 2000 (11.07.00), Page 2 (Family: none)	1-5									
<input type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/> See patent family annex.											
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Date of the actual completion of the international search 17 May, 2002 (17.05.02)		Date of mailing of the international search report 04 June, 2002 (04.06.02)									
Name and mailing address of the ISA/ Japanese Patent Office		Authorized officer  Telephone No.									
Facsimile No.											

Form PCT/ISA/210 (second sheet) (July 1998)