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(54) Title: METHOD FOR MANUFACTURING A HIGH STRENGTH STEEL SHEET HAVING IMPROVED FORMABILITY AND DUCTILITY AND SHEET OBTAINED

(57) Abstract: A method for manufacturing a high-strength sheet having improved formability and ductility accord which the chemical composition of the steel contains, in percent by weight: $0.25\% < C < 0.4\%$ $2.3\% \leq Mn \leq 3.5\%$ $2.3\% \leq Si \leq 3\%$ $Al \leq 0.040\%$ the reminder being Fe and unavoidable impurities, the method comprising the steps of annealing a rolled sheet made of said steel by soaking it at an annealing temperature AT higher than the Ac_3 transformation point of the steel, quenching the sheet by cooling it down to a quenching temperature QT between $M_s - 65^\circ C$ and $M_s - 115^\circ C$, being in order to obtain a structure containing at least 65 % of martensite and at least 15% of residual austenite, the sum of the ferrite and bainite contents being less than 10%, M_s being the M_s transformation point of the steel according to the Andrews formula, heating the sheet up to an overaging temperature PT between $360^\circ C$ and $500^\circ C$ and maintaining it at said temperature for a time Pt between 300 s and 600 s, and cooling the sheet down to the ambient temperature. Sheet obtained



METHOD FOR MANUFACTURING A HIGH STRENGTH STEEL SHEET HAVING IMPROVED FORMABILITY AND DUCTILITY AND SHEET OBTAINED

The present invention relates to high-strength steel sheets excellent in formability,
5 ductility and strength and to a method for producing the same.

To manufacture various equipments such as automotive parts, trailers, trucks and so
on, high strength steel sheets, made of steels such as DP (dual phase) or TRIP
(transformation induced plasticity) steels are used.

In order to reduce the weight of the equipments, which is very advisable in order to
10 reduce the energy consumption, it is desirable to have steels having better mechanical
properties such as yield strength or tensile strength. But such steels should have a good
formability and a good ductility.

For this purpose it was proposed to use steels containing about 0.2% C, 2.5% Mn,
1.5% Si and having a structure consisting of martensite and retained austenite. The
15 sheets are produced on continuous annealing lines by a heat treatment consisting on an
annealing, an interrupted quenching and an overaging. The purpose of the overaging is to
generate a carbon enrichment of the retained austenite by transfer from the martensite in
order to increase the stability of the retained austenite. In these steels, the Mn content
remains always less than 3.5%. Although with such steels it is possible to obtain
20 interesting properties, it remains a clear objective to obtain retained austenite having a
better stability in order to obtain better characteristics. But the ductility which is necessary
to have a good formability should remain at a high level, especially a very good stretch
flangeability is very helpful. More specifically, it is desirable to have a sheet having a yield
strength higher than 1050 MPa, an uniform elongation of at least 10% and a stretch
25 flangeability corresponding to a hole expansion ratio HER measured according to ISO
standard 16630:2009 of at least 25. In this respect, it should be noted that, due to the
differences between the methods of measure, the hole expansion ratio HER measured
according to the ISO standard 16630:2009 is not comparable to the hole expansion ratio λ
measured according to JFS T 1001 (Japan Iron and Steel Federation standard).

30 For this purpose, the invention relates to a method for manufacturing a high-strength
steel sheet having an improved formability according to which the chemical composition of
the steel contains, in percent by weight:

$$0.25\% < C \leq 0.4\%$$

$$2.3\% \leq Mn \leq 3.5\%$$

$$2.3\% \leq Si \leq 3.0\%$$

$$Al \leq 0.040\%$$

the reminder being Fe and unavoidable impurities, the method comprising the steps of:

- hot rolling a sheet made of said steel to obtain a hot rolled steel sheet,
- batch annealing said hot rolled steel sheet between 400°C and 700°C for a time
5 between 300 seconds to 10 hours,
- cold rolling said hot rolled and batch annealed steel sheet to obtain a cold rolled steel sheet,
- annealing said cold rolled steel sheet by soaking it at an annealing temperature AT higher than the transformation point of the steel $Ac_3 + 20^\circ\text{C}$, and less than 1100°C,
- 10 - quenching the sheet by cooling it down to a quenching temperature QT between $M_s - 65^\circ\text{C}$ and $M_s - 115^\circ\text{C}$, M_s being the M_s transformation point of the steel according to the Andrews formula, in order to obtain a structure containing at least 65% and preferably more than 75% of martensite and at least 15% of residual austenite, the sum of ferrite and bainite being less than 10%,
- 15 - heating the sheet up to an overaging temperature PT between 360°C and 500°C and maintaining it at said temperature for a time Pt between 300 s and 600 s and,
- cooling the sheet down to the room temperature.

Preferably, the chemical composition of the steel contains:

$$0.25\% < C \leq 0.35\%$$

$$2.4\% \leq \text{Mn} \leq 2.7\%$$

$$2.3\% \leq \text{Si} \leq 2.5\%$$

$$\text{Al} \leq 0.040\%$$

and the annealing temperature AT is greater than 860°C and less than 950°C, the quenching temperature QT is between 200°C and 260°C and the overaging temperature
25 is between 370°C and 430°C.

Optionally, the sheet can be furthered coated for example by hot dip coating with or without alloying.

The invention also relates to a high-tensile steel sheet made of steel which chemical composition contains in percent by weight:

$$0.25\% < C \leq 0.4\%$$

$$2.3\% \leq \text{Mn} \leq 3.5\%$$

$$2.3\% \leq \text{Si} \leq 3\%$$

$$\text{Al} \leq 0.040\%$$

the reminder being Fe and unavoidable impurities, the steel having a structure containing
35 more than 65% and preferably more than 75% of martensite, at least 15% of retained austenite, and less than 10% of the sum ferrite and bainite.

Preferably, the chemical composition of the steel contains:

$$0.25\% < C \leq 0.35\%$$

$$2.4\% \leq Mn \leq 2.7\%$$

$$2.3\% \leq Si \leq 2.5\%$$

$$Al \leq 0.040\%$$

The yield strength YS can be greater than or equal to 1050 MPa, the tensile strength greater than or equal to 1300 MPa, the uniform elongation UE greater than or equal to 10%, the total elongation greater than or equal to 13%, and the hole expansion ratio HER greater than or equal to 25%. Generally, the tensile strength is less than 2000 MPa.

Optionally, at least one face of the sheet is coated.

The invention will now be described in details and illustrated by examples without introducing limitations.

The composition of the steel according to the invention comprises, in weight percent:

- $0.25\% < C \leq 0.4\%$ and preferably $0.25\% < C \leq 0.35\%$ in order to obtain a satisfactory strength and to improve the stability of the retained austenite. If the carbon content is too high, the weldability is reduced.

- $2.3\% \leq Mn \leq 3.5\%$. The Mn content is higher than 2.3% and preferably higher than 2.4% in order to have a sufficient hardenability in order to be able to obtain a structure comprising at least 75% of martensite and to have a tensile strength of at least 1100 Mpa. But, the manganese content must remain less than 3.5%, and preferably less than 2.7% in order not to have too much segregation which is detrimental for the stretch flangeability.

- $Si \geq 2.3\%$, and $Si \leq 3\%$ and preferably $Si \leq 2.5\%$. Silicon is useful to stabilize the austenite, to provide solid solution strengthening and to delay the formation of carbides during carbon redistribution from martensite to austenite during overaging. But at a too high silicon content, silicon oxides will form at the surface of the sheet which is detrimental to coatability.

The reminder is Fe and impurities resulting from the melting. Such impurities include N, S, P and residual elements such as Cr, Ni, Mo, Cu, B and Al.

Usually, the N content remains less than 0.01%, the S content less than 0.01%, the P content less than 0.02%, the Cr content less than 0.1%, the Ni content less than 0.1%, the Mo content less than 0.05%, the Cu content less than 0.2%, the B content less than 0.0010% and the Al content less than 0.02%. However, it must be noted that Al may be added in order to deoxidize the steel. In this case, its content can reach 0.04%. Moreover, Al can form small precipitates of AlN which can be used to limit the austenitic grain growth during annealing.

No micro-alloying, such as Ti, V, and Nb, is targeted in the steel according to the invention. Such elements contents are limited individually to 0.050%, preferably, the sum of Nb, Ti, V is limited to 0.1 %.

Hot rolled sheet having a thickness between 2 and 5 mm can be produced in a known manner for this steel. As an example, the reheating temperature before rolling can be between 1200°C and 1280°C, preferably about 1250°C, the finish rolling temperature is preferably less than 850°C, the start cooling temperature less than 800°C, the stop cooling temperature between 570°C and 590°C and the coiling has to be made between 540°C and 580°C. After hot rolling, the sheet is batch annealed at a temperature between 400°C and 700°C for 300 seconds to 10 hours. The hot rolled sheet can be pickled and cold rolled to obtain a cold rolled sheet having a thickness between 0.5 mm and 2 mm.

Then, the sheet is heat treated on a continuous annealing line.

Before the heat treatment, an optimum quenching temperature Q_{Top} is determined. This optimum quenching temperature is the temperature at which the quenching has to be stopped in order to obtain an optimum content of retained austenite.

To determine this theoretical optimal quenching temperature Q_{Top} , the Andrews and the Koistinen-Marburger relationships can be used. These relationships are:

$$M_s = 539 - 423 \times C - 30.4 \times Mn - 12.1 \times Cr - 7.5 \times Mo - 7.5 \times Si$$

and:

$$f_{\alpha'} = 1 - \exp \{0.0011 \times (M_s - Q_T)\}$$

$f_{\alpha'}$ is the proportion of martensite obtained during quenching at the temperature Q_T . To determine the proportion of residual austenite after overaging and cooling down to the room temperature after quenching down to the quenching temperature Q_T , it is assumed that, after quenching, the sheet is overaged at a temperature higher than Q_T during a time sufficient to cause a sufficient partitioning of carbon between the martensite and the austenite with as low as possible formation of ferrite and bainite. It is also assumed that, after overaging, the sheet is cooled down to the room temperature.

Those which are skilled in the art know how to calculate the proportion of residual austenite and the theoretical optimal quenching temperature Q_{Top} which is the quenching temperature for which the proportion of residual austenite is maximal.

The theoretical optimal quenching temperature Q_{Top} is not necessarily the quenching temperature Q_T at which the quench has to be stopped.

The purpose of the heat treatment is to obtain a structure consisting of at least 65% and preferably at least 75% of martensite and at least 15% and preferably at least 20% of retained austenite with as little as possible of ferrite or bainite. The sum of ferrite and bainite surface fraction is less than 10% and preferably less than 5%.

The proportions of martensite, ferrite and bainite are area fractions of these constituents. The proportion of residual austenite is measured by X-RAY diffraction. Those who are skilled in the art know how to determine these proportions.

To make this heat treatment, the sheet is annealed at an annealing temperature AT higher than the A_{c3} transformation point of the steel and preferably equal or higher than $A_{c3} + 20^{\circ}\text{C}$ in order to have a completely austenite structure but less than 1100°C and preferably less than 950°C in order not to coarsen too much the austenite grains.

When the annealing is finished, the steel is quenched by cooling down to a quenching temperature QT at which the cooling is stopped. Then the sheet is heated to an overaging temperature PT at which it is maintained during an overaging time Pt before being cooled down to the room temperature or to be hot dip coated with or without alloying such as galvanizing, galvannealing or coating by aluminum alloys. .

For the quenching, the cooling speed has to be sufficiently high to avoid the formation of ferrite or bainite. In this respect, a cooling speed greater than 10°C/s is enough.

The quenching temperature QT is less than the M_s transformation point of the steel to ensure that the obtained structure is martensite and retained austenite.

The quenching temperature QT is such that the retained austenite content is sufficient to obtain the desired properties.

In the present invention, the quenching temperature QT is preferably between $Q_{\text{Top}} + 45^{\circ}\text{C}$ and $Q_{\text{Top}} - 5^{\circ}\text{C}$. As, for the steel of the present invention, Q_{Top} is about equal to $M_s - 110^{\circ}\text{C}$, therefore the quenching temperature QT can be chosen between $M_s - 65^{\circ}\text{C}$ and $M_s - 115^{\circ}\text{C}$, M_s being the M_s calculated using the Andrews formula..

The overaging temperature PT is between 360°C and 500°C and the overaging time Pt is between 300 s and 600 s.

With such heat treatment, the obtained structure of the steel contains at least 65% and even more than 75% of martensite and at least 15% of residual austenite, the sum of the ferrite and bainite contents remaining less than 10%. The content of residual austenite can be even greater than 20% and the sum of ferrite and bainite can even be less than 5%.

The martensite, ferrite and bainite contents are fraction areas and the austenite content is measured by X-Ray diffraction.

Optionally, between the overaging and the cooling down to the ambient (or room) temperature, the sheet can be hot dip coated for example galvanized or galvannealed. For this, the temperature of the sheet at the end of the overaging is adjusted to the hot dip coating bath temperature, for example 470°C (+/- 10°C) for zinc coating.

For a steel having the following preferable composition: $0.25\% < C \leq 0.35\%$, $2.4\% \leq Mn \leq 2.7\%$, $2.3\% \leq Si \leq 2.5\%$, the reminder being Fe and impurities, the annealing temperature is preferably greater than 860°C and less than 950°C , the quenching temperature QT is preferably between 200°C and 260°C and the overaging temperature is preferably between 370°C and 430°C .

Such sheet has a yield YS strength greater than 1050 MPa, a tensile strength TS greater than 1300 MPa, a uniform elongation UE greater than or equal to 10% and a hole expansion ratio HER measured according to ISO standard 16630:2009, greater than or equal to 25%.

As example and comparison, three steels corresponding to casts H118, H117 and H115 were produced. The compositions, the transformation points and the theoretical optional temperatures of the steels are reported in table I.

Table I

Cast	C %	Si %	Mn %	P %	S %	Al %	Ac ₁ °C	Ac ₃ °C	Ms °C	QTop °C	γ %
H118	0.293	2.4	2.5	0.0015	0.0045	0.006	777	842	321	210	29
H117	0.297	1.5	2.5	0.0013	0.0044	0.006	750	810	326	215	29
H115	0.293	1.6	3.3	0.0013	0.0047	0.014	757	806	302	195	30

In this table, Ac₁ and Ac₃ temperatures are measured by dilatometry, Ms is the value calculated using the Andrews formula, QTop is the theoretical optimal quenching temperature and γ% is the calculated fraction of residual austenite corresponding to QTop.

The sheets having a thickness of 1 mm were obtained by hot and cold rolling.

For the hot rolling, the reheating temperature was 1250°C , the finish rolling temperature was less than 850°C , the start cooling temperature for the water spray cooling was less than 800°C , the stop cooling temperature was between 570°C and 590°C and the coiling was made at 560°C .

The hot rolled sheet whose thickness was 2.5 mm was batch annealed at a temperature of 550°C for 10h. The hot rolled sheet was then cold rolled to obtain a cold rolled sheet having a thickness of 1mm.

Twenty one samples were taken in the sheets and were heat treated. For each cast, some samples were annealed above Ac₃ and the others under Ac₃ for a time of about 180 s, then quenched down to quenching temperatures QT equal to QTop, QTop - 20°C , QTop + 20°C and QTop + 40°C , and were overaged at 30°C or 400°C for 100 s or 500 s.

The conditions of the heat treatments and the obtained results are reported in table II.

Table II

Ex	Cast	AT °C	QT °C	PT °C	Pt s	γ %	YS Mpa	TS Mpa	UE %	HER %
1	H118	880	190	400	500	18	1220	1390	5	
2	H118	880	210	400	500	18	1175	1385	10	29
3	H118	880	230	400	500	18	1100	1380	10.5	
4	H118	880	250	400	500	17	1050	1365	14	
5	H118	880	210	400	100	16	1170	1430	8	
6	H118	880	210	350	500	16	1175	1430	5	
7	H118	820	210	400	500	24	750	1420	14	
8	H117	840	195	400	500	14	1200	1340	5	
9	H117	840	215	400	500	14	1150	1310	6	32
10	H117	840	235	400	500	16	1110	1310	7-10	-
11	H117	840	255	400	500	16	1040	1275	9	
12	H117	840	215	400	100	16	1120	1330	5	
13	H117	840	215	350	500	9	1150	1360	2	
14	H117	790	215	400	500	13	940	1275	12	
15	H115	820	175	400	500	17	1040	1420	10	
16	H115	820	195	400	500	19	1200	1430	10	11
17	H115	820	215	400	500	18	915	1490	9.5	
18	H115	820	235	400	500	17	850	1550	6	
19	H115	820	195	400	100	20	1000	1500	10	
20	H115	820	195	350	500	19	1000	1500	10	
21	H115	760	195	400	500	20	710	1390	2	

5

In this table, $\gamma\%$ is the residual austenite contents as measured on the sample by X-Ray diffraction, YS is the yield strength, TS the tensile strength, UE the uniform elongation and HER the hole expansion ratio according the ISO standard.

Examples 8 to 21 show that with casts H115 and H117, the desired results are not obtained.

10

Example 2, 3 and 4 shows that a yield strength of more than 1050 MPa, a tensile strength of more than 1300 MPa, a uniform elongation of at least 10% and can be obtained with cast H118, with an annealing temperature higher than A_{c3} , a quenching temperature QT between $M_s - 65^{\circ}\text{C}$ and $M_s - 115^{\circ}\text{C}$. equal to the optimal quenching temperature QTop, an averaging temperature of about 400°C and an overaging time of about 500 s. Example 2 shows that a hole expansion ratio of more than 25% can be obtained.

But the example 1 shows that if the quenching temperature is too low, at least the uniform elongation is not sufficient.

Examples 5, 6 and 7 show that, if the overaging time is too low, or the overaging temperature is too low or if the annealing temperature is under A_{c3} , the desired results are not obtained.

The sheet which is described above is not coated. But it is clear that the sheet may be coated by any means i.e. by hot dip coating, by electro-coating, by vacuum coating such as JVD or PVD and so on. When the sheet is hot dip coated, the coating can be galvanizing with or without alloying (galvanealing). In these cases, the heat treatment corresponding to the hot dip and eventually to the alloying which are made before cooling the sheet down to the ambient temperature have to be considered. Those which are skilled in the art know how to do that, for example by tests, in order to optimize the overaging temperature and time. In this case, at least one face of the sheet can be coated and more specifically metallic coated.

CLAIMS

1.- A method for manufacturing a high-strength steel sheet having improved formability and ductility according to which the chemical composition of the steel contains,
5 in percent by weight:

$$0.25\% < C \leq 0.4\%$$

$$2.3\% \leq Mn \leq 3.5\%$$

$$2.3\% \leq Si \leq 3\%$$

$$Al \leq 0.040\%$$

10 the remainder being Fe and unavoidable impurities, the method comprising the following successive steps:

- hot rolling a sheet made of said steel to obtain a hot rolled steel sheet,

- batch annealing said hot rolled steel sheet between 400°C and 700°C for a time between 300 seconds to 10 hours,

15 - cold rolling said hot rolled and batch annealed steel sheet to obtain a cold rolled steel sheet,

- annealing said cold rolled steel sheet by soaking it at an annealing temperature AT higher than the transformation point of the steel $Ac_3 + 20^\circ C$, and less than 1100°C,

20 - quenching the sheet by cooling it down to a quenching temperature QT between the $Ms - 65^\circ C$ and $Ms - 115^\circ C$ of the steel, in order to obtain a structure containing at least 65 % of martensite and at least 15% of residual austenite, the sum of ferrite and bainite being less than 10 %, Ms being the Ms transformation point of the steel according to the Andrews formula.

25 - heating the sheet up to an overaging temperature PT between 360°C and 500°C and maintaining it at said temperature for a time Pt between 300 s and 600 s and,

- cooling the sheet down to the room temperature.

30 2 – The method according to claim 1, characterized in that the sheet is cooled to the quenching temperature QT in order to obtain a structure containing at least 75 % of martensite.

3.- The method according to claim 1 or claim 2, characterized in that the chemical composition of the steel contains:

$$0.25\% < C \leq 0.35\%$$

$$2.4\% \leq Mn \leq 2.7\%$$

$$2.3\% \leq Si \leq 2.5\%$$

$$\text{Al} \leq 0.040\%$$

and in that the annealing temperature AT is greater than 860°C and less than 950°C, the quenching temperature QT is between 200°C and 260°C the overaging temperature PT is between 370°C and 430°C.

5

4.- The method according to any one of claims 1 to 3, characterized in that the sheet is further coated.

10

5.- A high-tensile steel sheet made of steel which chemical composition contains in percent by weight:

$$0.25\% < \text{C} \leq 0.4\%$$

$$2.3\% \leq \text{Mn} \leq 3.5\%$$

$$2.3\% \leq \text{Si} \leq 3\%$$

$$\text{Al} \leq 0.040\%$$

15

the reminder being Fe and unavoidable impurities, the steel having a structure containing more than 65 % of martensite, more than 15% of retained austenite, and less than 10% of the sum of ferrite and bainite.

20

6 - The high-tensile steel sheet according to claim 5, characterized in that the structure contains at least 75 % of martensite.

7.- The high-tensile steel sheet according to claim 5 or 6, characterized in that the chemical composition of the steel contains:

25

$$0.25\% < \text{C} \leq 0.35 \%$$

$$2.4\% \leq \text{Mn} \leq 2.7\%$$

$$2.3\% \leq \text{Si} \leq 2.5\%$$

$$\text{Al} \leq 0.040\%$$

30

the remainder being Fe and unavoidable impurities, and characterized in that the yield strength YS is greater than or equal to 1050 MPa, the tensile strength is greater than or equal to 1300 MPa the uniform elongation UE is greater than or equal to 10%, and the hole expansion ratio HER is greater than or equal to 25%.

8.- The high-tensile sheet according to claim 5, 6 or 7, characterized in that at least one face of the sheet is coated.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER

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ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

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C21D C22C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data, CHEM ABS Data, COMPENDEX, INSPEC

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X A A A	EP 2 660 345 A2 (POSCO [KR]) 6 November 2013 (2013-11-06) paragraphs [0001], [0023]-[0028], [0035], [0036], [0040]-[0042], [0045], [0047]-[0050], [0052]; claims 1, 3-5, 7, 11, 13-14; figure 1; tables 1-3 ----- EP 2 325 346 A1 (JFE STEEL CORP [JP]) 25 May 2011 (2011-05-25) the whole document ----- EP 2 546 375 A1 (JFE STEEL CORP [JP]) 16 January 2013 (2013-01-16) the whole document ----- -/--	1,2,4-6, 8 3,7 1-8 1-8



Further documents are listed in the continuation of Box C.



See patent family annex.

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INTERNATIONAL SEARCH REPORT

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C(Continuation). 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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