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(71) Applicant: **ARCELORMITTAL** [LU/LU]; 24-26, Boulevard d'Avranches, 1160 Luxembourg (LU).

(72) Inventors: **HOUYOUX, Christophe**; 15, rue du Marais, 6740 Etalle (BE). **RINALDI, Valérie**; 17 place Winston Churchill, 4056 Esch-Sur-Alzette (LU). **DONNAY, Boris**; 19 Rue Des Six Bonniers, 4550 Nandrin (BE). **WEBBER, Liudmila**; 6Bis rue Drogon, 57970 Yutz (FR).

(74) Agent: **PLAISANT, Sophie**; ArcelorMittal France, Research & Development, Intellectual Property, 6 rue André Campra, 93200 Saint-Denis (FR).

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(54) Title: STEEL SECTION HAVING A THICKNESS OF AT LEAST 100MM AND METHOD OF MANUFACTURING THE SAME

(57) Abstract: The invention deals with a steel section, comprising a web central portion connected on each side to a flange portion having a thickness of at least 100mm, such steel section having a composition comprising, in weight percentage: C : 0.06 - 0.16 % Mn : 1.10 - 2.00 % Si : 0.10 - 0.40 % Cu : 0.001 - 0.50 % Ni : 0.001 - 0.30 % Cr : 0.001 - 0.50 % Mo : 0.001 - 0.20 % V : 0.06 - 0.12 % N : 0.0050% - 0.0200 % Al ≤ 0.040 % P ≤ 0.040 % S ≤ 0.030 % and comprising optionally one or more of the following elements, in weight percentage: Ti < 0.005 % Nb ≤ 0.05 % the reminder being iron and impurities resulting from elaboration, and said steel section microstructure including at least one kind of vanadium precipitates possibly comprising also one or more metal chosen among chromium, manganese and iron, said precipitates being chosen among nitrides, carbides, carbo-nitrides or any combination of them, more than 70% of such precipitates having a mean diameter below 6 nm. It also deals with a manufacturing method thereof.



Steel section having a thickness of at least 100mm and method of manufacturing the same

The present invention deals with a steel section comprising a web
5 central portion connected on each side to a flange portion having a thickness above 100mm. The steel section according to the invention is particularly well suited for the manufacture of columns for high-rise buildings, long span, transfer and belt trusses, outriggers and bridge girders.

The development of new modern structural steel grades is always
10 driven by the users' requirements towards higher mechanical properties such as yield strength and toughness, as well as excellent technological properties, ensuring an efficient fabrication technology at workshop and on site.

The purpose of the invention therefore is to provide a steel heavy section reaching a high yield strength of at least 485 MPa and a high tensile
15 strength of at least 580 MPa with excellent weldability.

In practice of structural steel manufacturing, it is known that in order to improve strength and toughness it is preferable to refine the structure through hot rolling at lower temperatures or to add some alloying elements for austenite grain refining. Both solutions are not sufficient for heavy structural
20 steel manufacturing, because in case of lower hot rolling temperatures the overheating of the rolls is inevitable. At the same time, when the alloyed elements are added in high amounts, the weldability of the steel deteriorates.

The object of the present invention is achieved by providing a steel heavy section according to claim 1. The steel heavy section can also comprise
25 characteristics of claims 2 to 12. Another object is achieved by providing the method according to claim 13.

Other characteristics and advantages of the invention will become apparent from the following detailed description of the invention and the drawings :

30 - Figure 1 : shows an electron micrograph illustrating randomly distributed precipitates in the core of the flange of the heavy section,

- Figure 2 : shows an electron micrograph illustrating precipitates, arranged in regularly spaced bands.

All compositional percentages are given in weight percent (wt.%),
5 unless indicated otherwise. Regarding the chemical composition of the steel, carbon plays an important role in the formation of the microstructure and reaching of the targeted mechanical properties. Its main role is to provide strengthening through hardening of the martensite/bainite phases but also through formation of carbides and/or carbo-nitrides of metallic elements of the
10 steel. The carbon content of the grade according to the invention is between 0.06 and 0.16 % weight. Carbon content below 0.06% will not result in a sufficient level of mechanical resistance, leading to yield strengths value below 485 MPa. On the opposite, carbon contents above 0.16% would result in reducing ductility and the weldability of the steel. Preferably, the carbon
15 content is between 0.08 and 0.14 %, so as to obtain sufficient strength and weldability.

Manganese is an element which increases hardenability. The manganese content of the grade according to the invention is between 1.10 and 2.00 %. Manganese content below 1.10 % will not result in a sufficient
20 level of mechanical resistance. On the opposite, manganese content above 2.00 % would result in decreased weldability or would promote the formation of hard martensite-austenite constituents, also negatively impacting the toughness of the steel.

Silicon is a deoxidizing element and contributes to improving strength.
25 Silicon content below 0.10% will not result in a sufficient level of mechanical resistance nor a good deoxidation. On the opposite, silicon contents above 0.40% would result in the formation of oxides, reducing welding properties of the steel.

Copper is an element contributing to improving the strength of the steel
30 by hardenability improvement and precipitation strengthening. Copper content below 0.001% will not result in a sufficient level of mechanical resistance. On the opposite, copper contents above 0.50% would result in increasing the carbon equivalent and thus deteriorating the weldability or impacting the hot

shortness of the steel during hot deformation, caused by penetration of the Cu-enriched phase into grain boundaries.

Nickel is an element contributing to improving the strength and toughness of the steel. Nickel content below 0.001% will not result in a sufficient level of mechanical resistance. On the opposite, nickel contents above 0.30% would lead to high alloying costs.

Chromium is an element contributing to improving the strength of the steel by improving hardenability through solution hardening but also through precipitation hardening. Chromium content below 0.001% will not result in a sufficient level of mechanical resistance. On the opposite, chromium contents above 0.50% would result in generating coarse chromium carbides or carbo-nitrides that may deteriorate the toughness of the steel

Molybdenum is an element contributing to improving the strength of the steel by improving hardenability. Molybdenum content below 0.001% will not result in a sufficient level of mechanical resistance. On the opposite, molybdenum contents above 0.20% would result in reducing the toughness of the steel.

Vanadium is an important element that is used to achieve hardening and strengthening by precipitation of nitrides, carbo-nitrides or carbides but also through grain refining. The formation of vanadium precipitation limits the austenite grain coarsening, by resulting in ferrite grain decrease and improved strength by precipitation in ferrite phase. Vanadium would also prevent the chromium and manganese migration in the cementite, resulting in their application in small precipitation formation. Vanadium content below 0.06 % will not result in a sufficient level of mechanical resistance. On the opposite, vanadium contents above 0.12 % would result in a risk that an excessive precipitation may cause a reduction in toughness, which has to be avoided. In a preferred embodiment, vanadium addition is limited to 0.09% to improve further the toughness of the steel.

Nitrogen is an important element to form nitrides and carbo-nitrides of metallic elements like vanadium , niobium aluminum and titanium . Their size, distribution density and stability have a significant effect to mechanical strengthening. Nitrogen content below 0.0050% will not result in a sufficient

level precipitation and grain size control. To further improve those properties, a minimum level of 0.0060%, or even of 0.0070% or even better of 0.0080% is preferred. On the opposite, nitrogen contents above 0.0200 % would result in the presence of free nitrogen in the steel, which is known as having a negative impact on toughness in the Heat Affected Zone after welding.

During hot rolling, part of the vanadium will combine with nitrogen in order to form VN particles for austenite grain boundaries pinning. The remaining vanadium, in solution, will then precipitate in form of fine precipitates during cooling of the steel, thus making an important contribution to final strength. The inventors have found that the precipitation strengthening can be enhanced by optimizing the vanadium to nitrogen ratio in the steels section to approach the stoichiometric ratio of 4:1. In a preferred embodiment, the ratio of V to N is comprised between 2.5 and 7, and even comprised between 3 and 5.

Aluminium can be added in the steel for deoxidizing effect and removing of the oxygen from the steel. If other deoxidizing elements are added in the steel, the aluminum content is 0.005% and lower. Otherwise, the aluminum content is between 0.005% and 0.040%. If the aluminum content is too high, the formation of AlN will occur in preference to VN, and AlN being bigger in size than VN, it will be not as efficient for pinning of austenite grain boundaries as VN.

Sulfur and phosphorus are impurities that embrittle the grain boundaries and lead to the formation of center and micro-segregation. Their respective contents must not exceed 0.030 and 0.040% so as to maintain sufficient hot ductility and to avoid deterioration in welding properties.

Niobium is an element that may optionally be used to achieve hardening and strengthening by precipitation of nitrides, carbo-nitrides or carbides. It suppresses the growth of austenite grains during rolling, by refining them, thus resulting in improvement of strength and low-temperature toughness. However, when its amount is above 0.05%, could deteriorate toughness in the Heat Affected Zone due to martensite hardening. On the other hand, when niobium amount is 0.05% and higher, it will pin to available nitrogen and thus impairing nitrogen from forming vanadium precipitates that assures the strengthening of the ductile core of the section.

Titanium is an element that may optionally be used to achieve hardening and strengthening by precipitation of nitrides, carbo-nitrides or carbides. However, when its amount is above or equal to 0.005%, there is a risk of TiN formation rather than VN. Moreover, TiN being cuboids particles
5 may react as stress concentrators thus negatively impacting the toughness and fatigue properties of the steel. In a preferred embodiment, the maximum amount of titanium is set to 0.003% and even to 0.001%.

In a preferred embodiment, the carbon, manganese, chromium, molybdenum, vanadium, nickel and copper contents of the grade are such that
10
$$0.4 \leq \text{CEV} \leq 0.6$$
with
$$\text{CEV} = \text{C} + \text{Mn}/6 + (\text{Cr} + \text{Mo} + \text{V})/5 + (\text{Ni} + \text{Cu})/15$$

Respecting these values ensures that the hardenability of the steel section will be in suitable ranges through sufficient formation of bainite, while
15 maintaining a good weldability of the steel sections. The reduced carbon equivalent allows avoiding weld processing steps such as preheating (when acceptable) and also results in reduction of fabrication costs. In a preferred embodiment, $\text{CEV} \leq 0.5\%$.

20 The steel section comprises a web central portion connected on each side to a flange portion.

The thickness of the flange of the steel section according to the invention is set above 100 mm, allowing the use of such beam for high-rise building structures, notably. Its thickness is preferably below 140 mm as a
25 sufficient cooling rate to ensure the requested tensile and toughness properties is difficult to obtain.

According to the invention, the web and the flanges of the heavy section are composed of a hardened zone, resulting from the water cooling of the surface and a non-hardened zone, in the core of the product. Each zone of the
30 steel section can have a specific microstructure that can include one or more phases among tempered martensite, bainite, ferrite and pearlite. Ferrite can be present under the form of acicular ferrite or of regular ferrite.

The microstructure of each zone depends on the steel section thickness and on the thermal path it is submitted to.

In a preferred embodiment, the microstructure of the flanges portions include, from surface to core, a first zone comprising tempered martensite and possibly bainite and a second zone comprising ferrite and pearlite.

The first zone can, for example, extend up to 10 mm under the surface of the flange portion.

An essential characteristic of the invention is the presence, in the steel section microstructure, of at least one kind of vanadium precipitates possibly comprising also one or more metal chosen among chromium, manganese and iron, said precipitates being chosen among nitrides, carbides, carbo-nitrides or any combination of them, more than 70% of such precipitates and preferably more than 80%, having a mean diameter below 6 nm. The mean diameter determination was done in the following way: the surface of each detected precipitate was measured and applied to the corresponding circle, from which the diameter was extracted, giving then the mean diameter size for all detected precipitates.

In a preferred embodiment, the mean density of those precipitates is of at least 500 precipitates per mm², preferably of at least 1000 precipitates per mm². Those precipitates have a beneficial effect on strength, known as being increased with precipitates size decrease and precipitates content increase.

Such precipitates are preferably present in the core zone of the flange of the section, mainly in the ferrite phase. At least 70% of such precipitates and preferably at least 80%, have a mean diameter below 6 nm. The reduced size of such precipitates increases their hardening effect and hence the tensile strength of the steel section.

In a preferred embodiment, two types of precipitates are preferably present in the core of the flange of the steel section:

- precipitates randomly distributed inside ferrite and
- precipitates arranged in regularly spaced bands, forming thus parallel sheets densely populated with particles.

The randomly distributed precipitated are bigger than the one arranged in regularly spaced bands.

In a preferred embodiment, such regularly spaced precipitates include at least vanadium and chromium.

In another preferred embodiment more than 80% of the randomly distributed precipitates have a mean diameter between 3.5 and 6nm. Such
5 precipitates preferably include at least vanadium, chromium and iron.

The steel section according to the invention can be produced by any appropriate manufacturing method and the man skilled in the art can define one. It is however advisable to use a process ending by an accelerated
10 cooling, in that case quenching and self-tempering of the surface layer after hot-rolling step.

The method according to the invention comprises the following steps:

- 15 - feeding a semi-product which composition is according to the invention
- reheating such semi-product at a temperature above 1000°C and hot rolling it with a final rolling temperature of at least 900°C, to obtain a hot rolled steel section,
- 20 - cooling the hot rolled steel section so as to produce martensitic and/or bainitic quenching of the surface layer of all or part of the product, the non-quenched portion of the rolled product remaining at a temperature high enough to make it possible to cause a self-tempering of the quenched surface layer of martensite and/or bainite and to transform the austenite into ferrite and carbides in the core
25 part of the section during the subsequent cooling, the maximum temperature of the tempered surface of the product after quenching being 450 to 650°C and even 550-650°C

The steel sections according to the present invention are preferably
30 produced through a method in which a semi product made of a steel according to the present invention having the composition described above, is cast, the cast input stock is heated to a temperature above 1000°C, preferably above 1050°C and more preferably above 1100°C or 1150°C or used directly at such

a temperature after casting, without intermediate cooling. Such temperatures allow full dissolution of vanadium carbonitrides, which will further participate in precipitation strengthening mechanism.

The final hot-rolling step is performed at a temperature above 850°C.

- 5 The end-of-rolling temperature is above or equal to 850° C in order to assure the austenite grains refining and thus the formation of a thinner microstructure after transformation, which is known to enhance the toughness and strength properties.

- 10 During hot-rolling, it is preferable to use managed combination of rolling steps and controlling the rolling temperature. The aim is to create fine grained microstructure by grain refinement during the subsequent recrystallization during rolling.

The hot-rolled product obtained by the process described above is then cooled using preferably a quenching and self-tempering process.

- 15 The so-called quenching and self-tempering process (QST) consists in subjecting a hot rolled steel section emerging from the finishing stand of the rolling mill to cooling by means of a fluid so as to produce martensitic and/or bainitic quenching of the surface layer of all or part of the product. Moreover, at the outlet of the fluid cooling zone, the non-quenched portion of the rolled
20 product is at a temperature high enough to permit, during subsequent air cooling, tempering of the surface layer of martensite and/or bainite to take place.

- The cooling fluid employed for carrying out the quenching and self tempering step is usually water with or without conventional additives, or
25 aqueous of mineral salts, for example. The fluid may be a mist, for example obtained by suspending water in a gas, or it may be a gas, such as steam.

From a practical view point, desired cooling of the rolled products depends on the cooling devices used, and on suitable choice of the length and the flow rate characteristics of the cooling means.

- 30 The dimensions of the product are known as well as the composition of the steel, and thus its continuous cooling transformation diagram, making it possible to determine the conditions to apply for an adequate treatment of the steel section, among which, the temperature at which martensite is formed and

the maximum time available for performing surface quenching to the desired depth.

Based on curves of the temperature gradients in the core and the skin of the rolled steel section, the amount of heat to be removed can be as well as the characteristics of the cooling devices and the flow rates of the fluid applied by the cooling devices.

To monitor the formation of the desired microstructures in the different zones of the steel section, the evolutions of the skin temperature of the steel section starting from the end of the martensitic and/or bainitic quenching are being measured. After quenching, the skin temperature rises while the temperature at the core continuously decreases after the section has emerged from the last stand of the rolling mill. The skin temperature and the core temperature in a given cross-section converge towards a time from where the two curves continue substantially parallel to one another. The skin temperature at this point is called the "equalization temperature".

Examples

Two grades, which compositions are gathered in table 1, were cast in semi-products and processed into steel sections following the process parameters gathered in table 2, going through heating, controlled hot rolling and subsequent water cooling, achieved by quenching and self-tempering.

Table 1 - Compositions

The tested compositions are gathered in the following table wherein the element content are expressed in thousands of weight percent:

Trial	C	Mn	Si	Cu	Ni	Cr	Mo	V	N	Ti	Nb	Al	P	S	CEV
1	82	1059	171	170	162	129	49	<u>34</u>	9.6	1	1	3	13	23	0.32
2	98	1559	191	193	117	122	35	74	16.2	1	1	13	17	29	0.43

Trial 1 is a comparative example and trial 2 is an example according to the invention.

Table 2 – Process parameters

Steel semi-products, as cast, were processed under the following conditions:

Trial	Flange thickness (mm)	Hot rolling		Quenching and self-tempering	
		Reheating T (°C)	Hot rolling finish T (°C)	Specific water flow (l/m ² /s)	Self-tempering Temperature (°C)
1	80	1150	868	46	640
2	125	1170	893	46	600

5

The resulting samples were then analyzed and the corresponding microstructure elements and mechanical properties were respectively gathered in table 3 and 4.

10 Table 3 – Microstructure and precipitates

The phase percentages of the microstructures of the obtained steel section were determined:

Trial	Hardened zone		Core zone		
	Tempered martensite	Bainite	Regular Ferrite	Acicular ferrite	Pearlite +Bainite
1	33.4 %	66.6 %	76.7 %	5.5 %	17.7 %
2	56.7 %	43.3 %	72.4 %	0.4 %	27.2 %

15

The phase percentages in both zones, especially in the core zone, of section n°1 are quite similar to section n° 2, showing that the impact of the vanadium precipitation strengthening is observed at smaller microstructural scale.

20 Precipitation analysis done by TEM examination of carbon extraction replicas taken from the core zone of the flange thickness of the section, showed the presence of vanadium precipitates. Fine precipitates analysis was performed through TEM thin foil method, which allowed quantifying the mean size and the density of the precipitates.

It was found that precipitates participating in mechanical strengthening of the section were located in the core zone of the steel sections, in particular inside the ferrite phase.

Figure 1 shows the vanadium precipitates mostly having spherical shape, with bigger or smaller size. The bigger size precipitates (typical size about 6 nm in diameter) were mostly randomly distributed. But the fine precipitates (typical size about 3 nm in diameter) were arranged in regularly spaced bands. It can be seen on figure 2 that the microstructure consists of parallel sheets densely populated with vanadium particles. The sheets appear with a regular spacing.

Regularly spaced precipitates

Trial	Precipitates characteristics		
	% of precipitates with a mean diameter below 6 nm	Mean density in ferrite (per mm ²)	Mean diameter (nm)
1	<u>0%</u>	<u>< 100</u>	-
2	98%	1213	3.2

15 Randomly distributed precipitates

Trial	Precipitates characteristics			Repartition of metallic elements in precipitates, %			
	% of precipitates with a mean diameter below 6 nm	Mean density in ferrite (per mm ²)	Mean diameter (nm)	V	Cr	Mn	Fe
1	<u>0%</u>	<u>< 100</u>	-	-	-	-	-
2	70%	880	5.7	66.6	21.9	4.2	9.3

Table 4 – Mechanical properties

Mechanical properties of the tested steel were determined and gathered in the following table:

Trial	Yield Strength (MPa)	Tensile strength (MPa)	CEV (%)
1	<u>398</u>	<u>450</u>	0.32
2	495	653	0.43

5

The examples show that the steel sections according to the invention are the only one to show all the targeted properties thanks to their specific composition and microstructures.

Steel sections according to the present invention show excellent values of high strength, toughness and good weldability, which is nowadays not easily achievable. With the steel grade as per the invention, design and construction teams involved in large-scale construction projects can benefit from more efficient structural solutions. The steel section's higher yield strength enables weight savings and lower transportation and fabrication costs than other commonly-used structural steel grades. And thus, the present invention makes an extremely significant contribution to construction industry.

10

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CLAIMS

1. A steel section, comprising a web central portion connected on each side to a flange portion having a thickness of at least 100mm, such steel section having a composition comprising, in weight percentage:

5	C : 0.06 - 0.16 %
	Mn : 1.10 - 2.00 %
	Si : 0.10 - 0.40 %
	Cu : 0.001 - 0.50 %
	Ni : 0.001 - 0.30 %
10	Cr : 0.001 - 0.50 %
	Mo : 0.001 - 0.20 %
	V : 0.06 - 0.12 %
	N : 0.0050% - 0.0200 %
	Al $\leq$ 0.040 %
15	P $\leq$ 0.040 %
	S $\leq$ 0.030 %

and comprising optionally one or more of the following elements, in weight percentage:

	Ti < 0.005 %
20	Nb $\leq$ 0.05 %

the reminder being iron and impurities resulting from elaboration, and said steel section microstructure including at least one kind of vanadium precipitates possibly comprising also one or more metal chosen among chromium, manganese and iron, said precipitates being chosen among

25 nitrides, carbides, carbo-nitrides or any combination of them, more than 70% of such precipitates having a mean diameter below 6 nm.

2. A steel section according to claim 1, wherein such section composition is such that the following relationship is fulfilled:

$$0.4 \leq \text{CEV} \leq 0.6$$

with
$$\text{CEV} = \text{C} + \text{Mn}/6 + (\text{Cr} + \text{Mo} + \text{V})/5 + (\text{Ni} + \text{Cu})/15$$

- 5 3. A steel section according to claim 1 or 2, wherein the ratio of vanadium to nitrogen amounts is between 2.5 and 7.
- 10 4. A steel section according to anyone of claims 1 to 3, wherein the microstructure of said flanges portions including, from surface to core, a hardened zone comprising tempered martensite and possibly bainite and a core zone comprising ferrite and pearlite.
- 15 5. A steel section according to anyone of claims 1 to 4, wherein such steel section includes portions having a mean density of said precipitates of at least 500 precipitates per mm².
6. A steel section according to anyone of claims 1 to 5, wherein at least part of such precipitates is arranged in regularly spaced bands.
- 20 7. A steel section according to claim 6, wherein more than 80% of such regularly spaced precipitates have a mean diameter below 3 nm.
8. A steel section according to claim 6 or 7, wherein such regularly spaced precipitates include at least vanadium and chromium.
- 25 9. A steel section according to anyone of claims 4 to 8, wherein at least part of such precipitates is randomly distributed in the ferrite phase, located in the core of the steel section.
- 30 10. A steel section according to claim 9, wherein more than 80% of such randomly distributed precipitates have a mean diameter between 3.5 and 6nm.

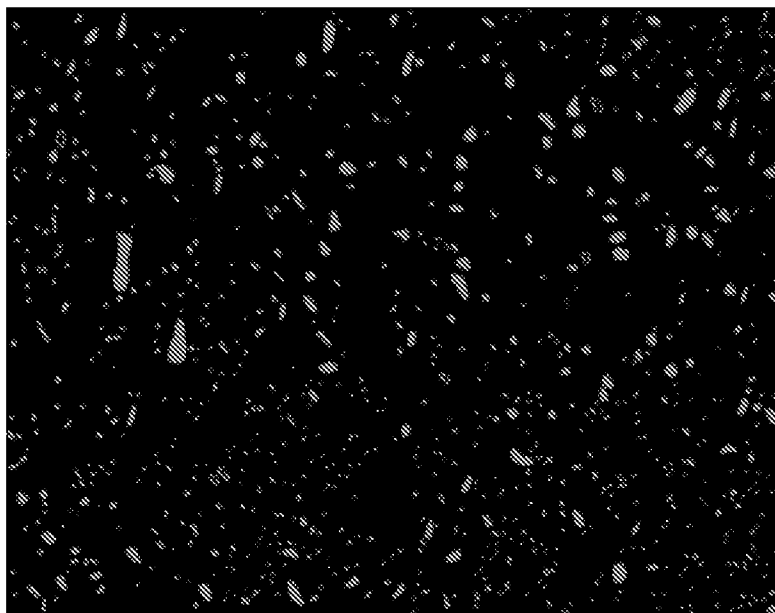
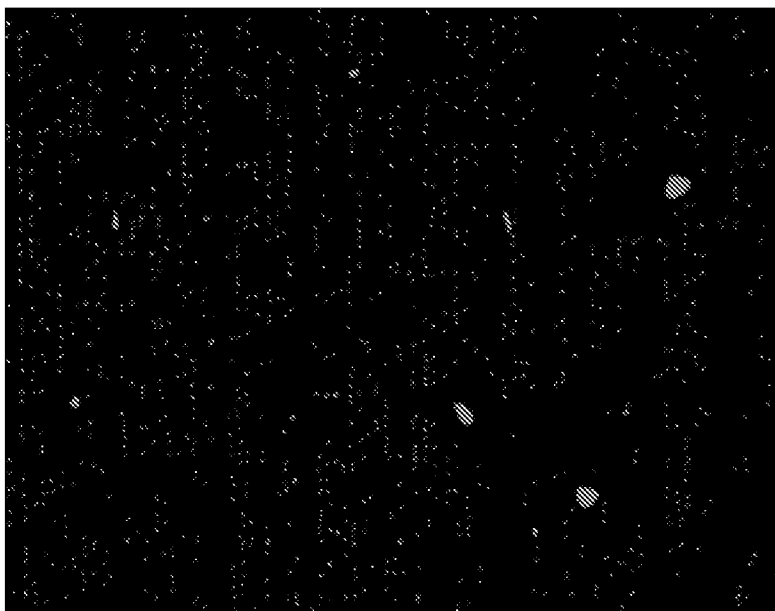
11.A steel section according to claim 10, wherein such randomly distributed precipitates include at least vanadium, chromium and iron.

5 12.A steel section according to anyone of claims 1 to 11, wherein said precipitates are located in said core zone.

13.A steel section according to anyone of claims 1 to 12, wherein said flange portions have a thickness of at most 140 mm.

10 14.A method of manufacturing of a steel section comprising the following steps:

- feeding a steel semi-product which composition is according to claims 1 to 3,
- 15 - reheating such steel semi-product at a temperature above 1000°C and hot rolling it with a final rolling temperature of at least 850°C, to obtain a hot rolled steel section,
- cooling the hot rolled steel section so as to produce martensitic and/or bainitic quenching of the surface layer of all or part of the product, the non-quenched portion of the rolled product remaining at
20 a temperature high enough to make it possible to cause a self-tempering of the quenched surface layer of martensite and/or bainite and to transform the austenite into ferrite and carbides in the core part of the section during the subsequent cooling, the maximum temperature of the tempered surface of the product after quenching
25 being 450 to 650°C.

Figure 1**Figure 2**

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2018/059909

A. CLASSIFICATION OF SUBJECT MATTER				
INV.	C22C38/00	B21B1/088	C21D8/02	C21D9/46
	C22C38/04	C22C38/06	C22C38/42	C22C38/44
	C22C38/48	C22C38/50	C22C38/58	C21D6/02
				C22C38/02
				C22C38/46
				C21D1/18
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)				
C22C B21B C21D E04C				
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				
C. DOCUMENTS CONSIDERED TO BE RELEVANT				
Category*	Citation of document, with indication, where appropriate, of the relevant passages			Relevant to claim No.
Y	JP H09 125140 A (KAWASAKI STEEL CO) 13 May 1997 (1997-05-13) paragraphs [0001] - [0003], [0008], [0028] - [0031], [0034], [0037] - [0040]; claims 1,7,8; examples G,H; tables 1,2			1-14
Y	EP 0 761 824 A2 (JFE STEEL CORP) 12 March 1997 (1997-03-12) page 1, lines 1-21 page 4, lines 25-44 page 5, line 48 - page 6, line 45 claims 1,10,11; examples G,H,I; tables 1,2 ----- -/--			1-14
☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.				
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