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(54) Titre : SUBSTRAT D'ACIER REVETU
(54) Title: A COATED STEEL SUBSTRATE

(57) Abrégé/Abstract:

The present invention relates to a coated steel substrate comprising a coating comprising nanographite having a lateral size between 1 and 60µm and a binder, wherein the steel substrate has the following compositions in weight percent: $0.31 \leq C \leq 1.2\%$, $0.1 \leq Si \leq 1.7\%$, $0.7 \leq Mn \leq 3.0\%$, $P \leq 0.01\%$, $S \leq 0.1\%$, $Cr < 0.5\%$, $Ni < 0.5\%$, $Mo < 0.1\%$, and on a purely optional basis, one or more elements such as $Nb \leq 0.05\%$, $B \leq 0.003\%$, $Ti \leq 0.06\%$, $Cu \leq 0.1\%$, $Co \leq 0.1\%$, $N \leq 0.01\%$, $V \leq 0.05\%$, the remainder of the composition being made of iron and inevitable impurities resulting from the elaboration; and a method for the manufacture of the coated steel substrate.

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(54) Title: A COATED STEEL SUBSTRATE

(57) Abstract: The present invention relates to a coated steel substrate comprising a coating comprising nanographite having a lateral size between 1 and 60µm and a binder, wherein the steel substrate has the following compositions in weight percent: $0.31 \leq C \leq 1.2\%$, $0.1 \leq Si \leq 1.7\%$, $0.7 \leq Mn \leq 3.0\%$, $P \leq 0.01\%$, $S \leq 0.1\%$, $Cr < 0.5\%$, $Ni < 0.5\%$, $Mo < 0.1\%$, and on a purely optional basis, one or more elements such as $Nb \leq 0.05\%$, $B \leq 0.003\%$, $Ti \leq 0.06\%$, $Cu \leq 0.1\%$, $Co \leq 0.1\%$, $N \leq 0.01\%$, $V \leq 0.05\%$, the remainder of the composition being made of iron and inevitable impurities resulting from the elaboration; and a method for the manufacture of the coated steel substrate.

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A coated steel substrate

The present invention relates to a steel substrate coated with a coating including nanographite, having a specific lateral size, and a binder, a method for the manufacture of this coated steel substrate. It is particularly well suited for steel industry.

In the steel route production, after the steel making step, the steel is casted in the continuous casting. Semi-products, such as slabs, billets or blooms, are thus obtained. Usually, the semi-products are reheated at high temperature in a reheating furnace to dissolve the precipitates formed during the continuous casting and to obtain a hot workability. They are then descaled and hot-rolled. However, during the reheating step, semi-products, especially having high carbon content, decarburized resulting in a change of mechanical properties. Indeed, during the reheating step, the semi-product can lose a large carbon content. For example, rails steels decarburize leading in a decrease the hardness of the steel. Consequently, mechanical properties at the rail head are low and mechanical properties of the whole rail head are heterogeneous. Thus, there is a need to find a way to prevent the decarburization of a steel substrate during the reheating.

The patent application CN101696328 discloses a protective coating for a surface of a steel piece in order to prevent the surface from oxidation and decarburization at high temperature and, improve hardness and abrasion resistance and ultimately increase the overall service life of the steel workpiece, for the case of oxidation and decarburization of a surface (substrate) of a steel workpiece at high temperature, and the surface oxidation decarburization under the oxidizing atmosphere during heat treatment, forging, hot rolling, roll forming heating, particularly for the case that the steel workpiece is easy to be oxidized and decarbonized at high temperature in a heat treatment, leading to reduction in carbon atoms and carbon content, and the change in the surface (substrate) microstructure results in a reduced hardness, a reduced abrasion resistance and a short overall service life.

In this patent, the coating has a composition of: graphite, water glass and surface penetrant, in which a volume ratio of the graphite to sodium silicate is 1: 3

to 1: 7, and the surface penetrant constitutes 0.05% to 0.15% by volume of the coating. Nevertheless, the tests were performed with low carbon steels including 25 (carbon steel) and HT300 (cast iron) and with very high alloy steels including 32CrMo and Mn13.

Thus, the purpose of the invention is to provide a hot steel product having a specific steel composition, wherein the decarburization is significantly reduced during the reheating step.

According to one aspect of the disclosure, there is provided a coated steel substrate comprises a coating having a thickness between 10 and 250 μ m, the coating comprising nanographite flakes having a lateral size between 1 and 60 μ m and a binder, wherein the steel substrate has the following composition in weight percent:

$$0.31 \leq C \leq 1.2\%,$$

$$0.1 \leq Si \leq 1.7\%,$$

$$0.7 \leq Mn \leq 3.0\%,$$

$$P \leq 0.01\%,$$

$$S \leq 0.1\%,$$

$$Cr \leq 0.5\%,$$

$$Ni \leq 0.5\%,$$

$$Mo \leq 0.1\%,$$

the remainder of the composition being made of iron and inevitable impurities resulting from elaboration.

The coated steel substrate may further have one or more of the following features:

- the composition further comprises in weight percent:

$$Nb \leq 0.05 \%,$$

$$B \leq 0.003\%,$$

$$Ti \leq 0.06\%,$$

$$Cu \leq 0.1\%,$$

$$Co \leq 0.1\%,$$

$$N \leq 0.01\%,$$

$$V \leq 0.05\%.$$

- the lateral size of the nanoparticles is between 20 and 55 μ m.

2a

- the lateral size of the nanoparticles is between 30 and 55µm.
- wherein the thickness of the coating is between 10 and 250µm.
- the steel substrate is a slab, a billet or a bloom.
- the binder is sodium silicate or the binder includes aluminum sulfate and an additive being alumina.
- the coating further comprises an organometallic compound.
- the organometallic compound includes Dipropylene glycol monomethyl ether ($\text{CH}_3\text{OC}_3\text{H}_6\text{OC}_3\text{H}_6\text{OH}$), 1,2-Ethanediol ($\text{HOCH}_2\text{CH}_2\text{OH}$) and 2-ethylhexanoic acid, manganese salt ($\text{C}_8\text{H}_{16}\text{MnO}_2$).

According to another aspect of the disclosure, there is provided a method for the manufacture of the coated steel substrate as described herein, comprises the successive following steps:

- A. providing a steel substrate as described herein,
- B. coating deposition using an aqueous mixture to form the coating as described herein,
- C. optionally, drying the coated steel substrate obtained in step B).

The method described herein may further have one or more of the following features:

- in step B), the deposition of the coating is performed by spin coating, spray coating, dip coating or brush coating.
- in step B), the aqueous mixture comprises from 1 to 60g/L of nanographite and from 150 to 250g/L of binder.
- in step B), wherein the aqueous mixture comprises nanographite comprising above 95% by weight of C.
- in step B), the aqueous mixture comprises nanographite comprising an amount of C equal or above to 99% by weight.
- in step B), the ratio in weight of nanographite with respect to binder is below or equal to 0.3.
- in step B), the aqueous mixture comprises an organometallic compound.

- in step B), the concentration of the organometallic compound is equal to or below 0.12wt. %.
- in step C), when a drying is applied, the drying is performed at a temperature between 50 and 150°C or at room temperature.
- in step C), when a drying is applied, the drying step is performed with hot air.
- in step C), when a drying is applied, the drying is performed during 5 to 60 minutes.

According to another aspect of the disclosure, there is provided a method for manufacture of a hot rolled steel product comprises the following successive steps:

- I. providing a coated steel substrate as described herein or obtained from the method as described herein,
- II. reheating the coated steel substrate in a reheating furnace at a temperature between 750 and 1300°C,
- III. descaling the reheated coated steel sheet obtained in step II) and
- IV. hot-rolling the descaled steel product.

The method described herein, may further have one or more of the following features:

- in step II), the reheating is performed at a temperature between 800 and 1300°C.
- in step III), the descaling is performed using water under pressure or the descaling is performed mechanically.
- in step III), the water pressure is between 100 and 150 bars.

To illustrate the invention, various embodiments and trials of non-limiting examples will be described, particularly with reference to the following Figure:

Figure 1 illustrates an example of coated steel substrate according to the present invention.

Figure 2 illustrates an example of a nanographite coating over a steel substrate according to the present invention.

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

The invention relates to a coated steel substrate comprising a coating comprising nanographite having a lateral size between 1 and 60 μ m and a binder, wherein the steel substrate has the following compositions in weight percent:

$$0.31 \leq C \leq 1.2\%,$$

$$0.1 \leq Si \leq 1.7\%,$$

$$0.7 \leq Mn \leq 3.0\%,$$

$$P \leq 0.01\%,$$

$$S \leq 0.1\%,$$

$$Cr \leq 0.5\%,$$

$$Ni \leq 0.5\%,$$

Mo $\leq$ 0.1%,
and on a purely optional basis, one or more elements such as

Nb $\leq$ 0.05 %,

B $\leq$ 0.003%,

5 Ti $\leq$ 0.06%,

Cu $\leq$ 0.1%,

Co $\leq$ 0.1%,

N $\leq$ 0.01%,

V $\leq$ 0.05%,

10 the remainder of the composition being made of iron and inevitable impurities
resulting from the elaboration.

Without willing to be bound by any theory, it seems that a coating comprising nanographite having a lateral size between 1 and 60 μ m and a binder on a steel substrate having the above specific steel composition reduces the
15 decarburization during the reheating of the coated steel substrate. The inventors have found that not only the steel composition but also the nature of coating plays an important role on the reduction or the elimination of steel decarburization during the heating treatment.

Indeed, It seems that there is a competition between oxidation and
20 decarburization kinetics during the reheating. For the above specific steel substrate (5), the formation of iron of the steel into scale reduces the decarburized layer. Additionally, as illustrated in Figure 1, it is believed that in the coating (1) nanographite flakes (2) having the specific lateral size are well dispersed into the binder (3) forming a tortuous path (4) allowing a carburization of the decarburized
25 areas. Indeed, it seems that there is a carbon restoration due to the presence of nanographite having the specific lateral size into the coating.

Regarding the chemical composition of the steel, preferably, the C amount is between 0.31 and 1.0% by weight.

Preferably, the Mn amount is between 0.9 and 2.5% and preferably
30 between 1.1 and 2.0% by weight.

Advantageously, the amount of Cr is below or equal to 0.3% by weight.

Preferably, the amount of Ni is below or equal to 0.1% by weight.

Advantageously, the amount of Mo is below or equal to 0.1%.

Figure 2 illustrates an example of nanographite flake according to the present invention. In this example, the lateral size means the highest length of the nanoplatelet through the X axis and the thickness means the height of the nanoplatelet through the Z axis. The width of the nanoplatelet is illustrated through the Y axis.

Preferably, the lateral size of the nanoparticles is between 20 and 55µm and more preferably between 30 and 55µm.

Preferably, the thickness of the coating is between 10 and 250µm. For example, the thickness of the coating is between 10 and 100µm or between 100 and 250µm.

Advantageously, the steel substrate is a slab, a billet or a bloom.

Preferably, the binder is sodium silicate or the binder includes aluminum sulfate and an additive being alumina. In this case, without willing to be bound by any theory, it seems that the coating according to the present invention better adheres on the steel substrate so that the steel substrate is even more protected. Thus, the risk of coating cracks and coating detachment, exposing the steel substrate to decarburization, is more prevented.

Preferably, the coating further comprises an organometallic compound. For example, the organometallic compound includes Dipropylene glycol monomethyl ether ($\text{CH}_3\text{OC}_3\text{H}_6\text{OC}_3\text{H}_6\text{OH}$), 1,2-Ethanediol ($\text{HOCH}_2\text{CH}_2\text{OH}$) and 2-ethylhexanoic acid, manganese salt ($\text{C}_8\text{H}_{16}\text{MnO}_2$). Indeed, without willing to be bound by any theory, it is believed that the organometallic compound allows for a fast curing of the coating avoiding a drying step at high temperature.

The invention also relates to a method for the manufacture of the coated steel substrate according to the present invention, comprising the successive following steps:

- A. The provision of a steel substrate having the above steel composition,
- B. The coating deposition using an aqueous mixture to form the coating and
- C. Optionally, the drying of the coated steel substrate obtained in step B).

Preferably, in step B), the deposition of the coating is performed by spin coating, spray coating, dip coating or brush coating.

Advantageously, in step B), the aqueous mixture comprises from 1 to 60g/L of nanographite and from 150 to 250g/L of binder. More preferably, the aqueous mixture comprises from 1 to 35g/L of nanographite.

Preferably, in step B), wherein the aqueous mixture comprises nanographite comprising above 95% and advantageously 99% by weight of C.

Advantageously, in step B), the ratio in weight of nanographite with respect to binder is below or equal to 0.3.

Preferably, in step B), the aqueous mixture comprises an organometallic compound. More preferably, the concentration of the organometallic compound is equal or below to 0.12wt.%. Indeed, without willing to be bound by any theory, it is believed that this concentration allows for an optimized coating without any curing or with a curing at room temperature.

In a preferred embodiment, the coating is dried in a step C). Without willing to be bound by any theory, it is believed that the drying step allows for an improvement of the coating adhesion. Indeed, since water evaporates, the binder becomes tackier and more viscous leading to a hardened condition. In a preferred embodiment, in step C), the drying is performed at room temperature or a temperature between 50 and 150°C and preferably between 80 and 120°C.

In another preferred embodiment, no drying step is performed.

Preferably, in step C), when a drying is applied, the drying step is performed with hot air.

Advantageously, in step C), when a drying is applied, the drying is performed during 5 to 60minutes and for example, between 15 and 45minutes.

The invention also relates to a method for manufacture of a Hot rolled steel product comprising the following successive steps:

- I. The provision of the coated steel substrate according to the present invention,
- II. The reheating of the coated steel substrate in a reheating furnace at a temperature between 750 and 1300°C,

III. The descaling of the reheated coated steel sheet obtained in step II) and

IV. The hot-rolling of the descaled steel product.

Preferably, in step I), the reheating is performed at a temperature between
5 800 and 1300°C, more preferably between 900 and 1300°C and advantageously between 1100 and 1300°C.

Advantageously, in step III), the descaling is performed using water under pressure. For example, the water pressure is between 100 and 150 bars. In another embodiment, the descaling is performed mechanically, for example, by
10 scratching or brushing the scale layer.

With the method according to the present invention, a hot rolled steel product wherein the surface is mainly not decarburized is obtained.

For example, after the hot-rolling, the hot product can be coiled, cold-rolled, annealed in an annealing furnace and also coated with a metallic coating.

Finally, the invention relates to the use of a hot rolled steel product obtainable from the method according to the present invention for the manufacture of a part of an automotive vehicle, a rail, a wire or a spring.
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The invention will now be explained in trials carried out for information only. They are not limiting.

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Examples:

In Examples, steels substrates having the following steel composition in weight percent were used:

Steel	C	Mn	Si	Cu	Cr	Ti	V	Mo	Ni
1	0.39	0.673	1.593	0.011	0.036	0.003	0.002	0.001	0.014
2	0.798	1.310	0.446	0.014	0.097	0.0014	0.0026	0.0018	0.016
3	0.901	0.309	0.244	0.017	0.215	0.002	0.002	0.001	0.019

25 Trial 1 was casted in the form of slab and Trials 2 and 3 were casted in the form of bloom.

Example 1: Decarburization test

For some Trials, steels were coated by spraying an aqueous mixture comprising 30g/L of nanographite having a lateral size between 35-50µm, a binder being Na₂SiO₃ (sodium silicate) and optionally an organometallic compound being DriCAT® onto the steel. Then, optionally, the coating was dried at room temperature or during 30 minutes at 100°C.

Then, uncoated steels and coated steels were reheated at 1250°C. After the reheating, the trials were analyzed by optical microscopy (OM). 0 means that almost no decarburized areas are present at the trial surface, i.e. almost no decarburization happened, during the reheating and 1 means that a lot of decarburized areas are present at the surface of the trial.

The results are in the following Table 1:

Trials	Steels	Coating	Curing after coating deposition	Reheating step		decarburization
				temperature (°C)	time	
1*	1	Aqueous mixture	30min at 100°C	1250	3h	0
2*	1	Aqueous mixture	30min at 100°C	1250	6h	0
3	1	-	-	1250	3h	1
4*	2	Aqueous mixture	30min at 100°C	1250	2h	0
5*	2	Aqueous mixture	30min at 100°C	1250	6h	0
6*	2	Aqueous mixture including DriCAT®	No curing	1250	6h	0
7*	2	Aqueous mixture including DriCAT®	Room temperature	1250	6h	0
8	2	-	-	1250	2h	1
9*	2	Aqueous mixture	30min at 100°C	1250	3h	0
10	2	-	-	1250	3h	1
11	3	Aqueous mixture	30min at 100°C	1250	3h	1

12	3	-	-	1250	3h	1
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*: according to the present invention.

For Trials according to the present invention, a very low amount of carbon was removed at the trial surface. On the contrary, for comparative Trials, a lot of decarburized areas were present allowing a change in the microstructure and therefore mechanical properties. Indeed, in the areas where there is a lot of carbon depletion, i.e. decarburized areas, ferrite is formed instead of pearlite.

Example 2: Microhardness test

In this case, after the reheating at 1250°C, some Trials were quenched in water to form martensite and the microhardness evolution from the hot steel product surface to a depth of 1500µm was determined by microhardness measurements. Indeed, when martensite is formed, the carbon content of the martensite is directly proportional to the amount of carbon in the microstructure.

Therefore, the higher the microhardness is, the higher the carbon content is.

The results are in the following Table 2:

Trials	Steels	Coating	Reheating step		Microhardness (HV)			
			temperature (°C)	time	100(µm)	500(µm)	1000(µm)	1500(µm)
4*	2	Aqueous mixture	1250	2h	840	840	840	840
8	2	-	1250	2h	<u>280</u>	<u>420</u>	<u>600</u>	<u>700</u>
9*	2	Aqueous mixture	1250	3h	820	840	900	900
10	2	-	1250	3h	<u>380</u>	<u>640</u>	<u>820</u>	<u>900</u>

*: according to the present invention.

The microhardness of Trials 4 and 8 clearly show that the decarburization was significantly reduced with the coated steel substrate according to the present invention compared to Trials 9 and 10.

CLAIMS

1. A coated steel substrate comprising a coating having a thickness between 10 and 250µm, the coating comprising nanographite flakes having a lateral size between 1 and 60µm and a binder, wherein the steel substrate has the following composition in weight percent:

$$0.31 \leq C \leq 1.2\%,$$

$$0.1 \leq Si \leq 1.7\%,$$

$$0.7 \leq Mn \leq 3.0\%,$$

$$P \leq 0.01\%,$$

$$S \leq 0.1\%,$$

$$Cr \leq 0.5\%,$$

$$Ni \leq 0.5\%,$$

$$Mo \leq 0.1\%,$$

the remainder of the composition being made of iron and inevitable impurities resulting from elaboration.

2. A coated steel substrate according to claim 1, wherein the composition further comprises in weight percent:

$$Nb \leq 0.05 \%,$$

$$B \leq 0.003\%,$$

$$Ti \leq 0.06\%,$$

$$Cu \leq 0.1\%,$$

$$Co \leq 0.1\%,$$

$$N \leq 0.01\%,$$

$$V \leq 0.05\%.$$

3. A coated steel substrate according to claim 1 or 2, wherein the lateral size of the nanoparticles is between 20 and 55µm.
4. A coated steel substrate according to claim 3, wherein the lateral size of the nanoparticles is between 30 and 55µm.

5. A coated steel substrate according to any one of claims 1 to 4, wherein the thickness of the coating is between 10 and 250 μ m.
6. A coated steel substrate according to any one of claims 1 to 5, wherein the steel substrate is a slab, a billet or a bloom.
7. A coated steel substrate according to any one of claims 1 to 6, wherein the binder is sodium silicate or the binder includes aluminum sulfate and an additive being alumina.
8. A coated steel substrate according to any one of claims 1 to 7, wherein the coating further comprises an organometallic compound.
9. A coated steel substrate according to claim 8, wherein the organometallic compound includes Dipropylene glycol monomethyl ether ($\text{CH}_3\text{OC}_3\text{H}_6\text{OC}_3\text{H}_6\text{OH}$), 1,2-Ethandiol ($\text{HOCH}_2\text{CH}_2\text{OH}$) and 2-ethylhexanoic acid, manganese salt ($\text{C}_8\text{H}_{16}\text{MnO}_2$).
10. A method for the manufacture of the coated steel substrate according to any one of claims 1 to 9, comprising the successive following steps:
 - A. providing a steel substrate having the composition as defined in claim 1 or 2,
 - B. coating deposition using an aqueous mixture to form the coating as defined in any one of claims 1 to 9,
 - C. Optionally, drying the coated steel substrate obtained in step B).
11. A method according to claim 10, wherein in step B), the deposition of the coating is performed by spin coating, spray coating, dip coating or brush coating.
12. A method according to claim 10 or 11, wherein in step B), the aqueous mixture comprises from 1 to 60g/L of nanographite and from 150 to 250g/L of binder.

13. A method according to any one of claims 10 to 12, wherein in step B), the aqueous mixture comprises nanographite comprising above 95% by weight of C.
14. A method according to claim 13, wherein in step B), the aqueous mixture comprises nanographite comprising an amount of C equal to or above 99% by weight.
15. A method according to any one of claims 10 to 14, wherein in step B), the ratio in weight of nanographite with respect to binder is below or equal to 0.3.
16. A method according to any one of claims 10 to 15, wherein in step B), the aqueous mixture comprises an organometallic compound.
17. A method according to claim 16, wherein in step B), the concentration of the organometallic compound is equal to or below 0.12wt.%.
18. A method according to any one of claims 10 to 17, wherein in step C), when a drying is applied, the drying is performed at a temperature between 50 and 150°C or at room temperature.
19. A method according to any one of claims 10 to 18, wherein in step C), when a drying is applied, the drying step is performed with hot air.
20. A method according to any one of claims 10 to 19, wherein in step C), when a drying is applied, the drying is performed during 5 to 60 minutes.
21. A method for manufacture of a hot rolled steel product comprising the following successive steps:
- I. providing a coated steel substrate according to any one of claims 1 to 9 or obtained from the method according to any one of claims 10 to 20,

- II. reheating the coated steel substrate in a reheating furnace at a temperature between 750 and 1300°C,
- III. descaling the reheated coated steel sheet obtained in step II) and
- IV. hot-rolling the descaled steel product.

22. A method according to claim 21, wherein in step II), the reheating is performed at a temperature between 800 and 1300°C.

23. A method according to claim 21 or 22, wherein in step III), the descaling is performed using water under pressure or the descaling is performed mechanically.

24. A method according to claim 23, wherein in step III), the water pressure is between 100 and 150 bars.

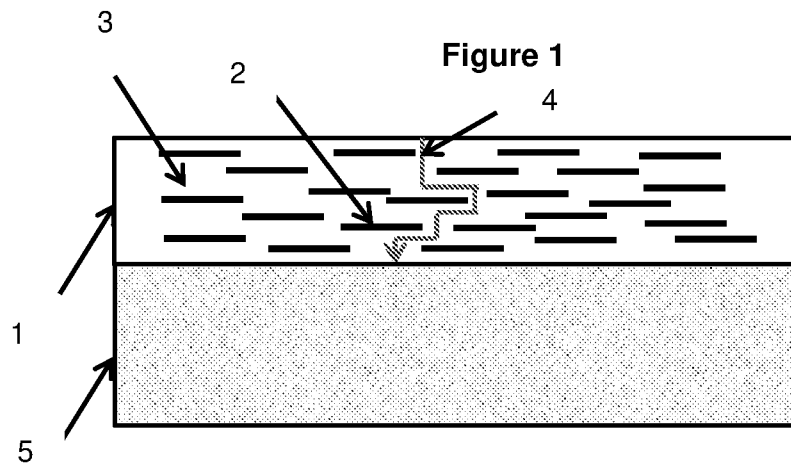


Figure 2

