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(54) Title: METHOD OF PRODUCING A COLD-ROLLED BAND OF DUAL-PHASE STEEL WITH A FERRITIC/MARTENSITIC STRUCTURE AND BAND THUS OBTAINED

(54) Titre : PROCÉDÉ DE FABRICATION D'UNE BANDE D'ACIER DUAL-PHASE A STRUCTURE FERRITO-MARTENSIQUE, LAMINÉE A FROID ET BANDE OBTENUE

(57) **Abstract:** The invention relates to a method of producing a cold-rolled band of dual-phase steel with a ferritic/martensitic structure. The inventive method consists in hot rolling a slab having a chemical composition which comprises, by weight,  $0.01\% \leq C \leq 0.1\%$ ,  $0.05\% \leq Mn \leq 1\%$ ,  $0.01\% \leq Cr \leq 1\%$ ,  $0.01\% \leq Si \leq 0.5\%$ ,  $0.001\% \leq P \leq 0.2\%$ ,  $0.01\% \leq Al \leq 0.1\%$ ,  $N \leq 0.01\%$ , the remainder being iron and impurities resulting from the preparation thereof. The method also comprises the following subsequent steps consisting in: hot winding the band obtained at a temperature of between 550 and 850 °C; cold rolling the band with a reduction ratio of between 60 and 90%; annealing the band continuously in the intercritical region; cooling said band to ambient temperature, in one or more steps, the rate of cooling between 600 °C and ambient temperature being between 100 °C/s and 1500 °C/s; and, optionally, tempering same at a temperature of less than 300 °C. The aforementioned annealing and cooling operations are performed such that the end band comprises between 1 and 15% martensite. The invention also relates to the steel band thus formed.

(57) **Abrégé :** L'invention concerne un procédé de fabrication d'une bande d'acier dualphase à structure ferrito-martensitaire, laminée à froid dans lequel on lamine à chaud une brame dont la composition chimique comprend, en poids,  $0,010\% \leq C \leq 0,100\%$ ,  $0,050\% \leq Mn \leq 1,0\%$ ,  $0,010\% \leq Cr \leq 1,0\%$ ,  $0,010\% \leq Si \leq 0,50\%$ ,  $0,001\% \leq P \leq 0,20\%$ ,  $0,010\% \leq Al \leq 0,10\%$ ,  $N \leq 0,010\%$  le reste étant du fer et des impuretés résultant de l'élaboration, ledit procédé comprenant ensuite les étapes consistant à - bobiner la bande à chaud obtenue à une température comprise entre 550 et 850°C, puis - à laminer à froid la bande avec un taux de réduction compris entre 60 et 90%, puis - à recuire la bande de façon continue dans le domaine intercritique, et - à la refroidir jusqu'à la température ambiante, en une ou plusieurs étapes, la vitesse de refroidissement entre 600°C et la température ambiante étant comprise entre 100°C/s et 1500°C/s, - et éventuellement à lui faire subir un revenu à une température inférieure à 300°C, les opérations de recuit et de refroidissement étant menées de telle sorte que la bande comprenne finalement de 1 à 15% de martensite, et la bande d'acier ainsi formée.

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**Process for producing a cold-rolled band of dual-phase steel with a ferritic/martensitic structure, and band thus obtained**

5

The present invention relates to a process for producing a cold-rolled ferritic/martensitic dual-phase steel strip and to a strip that can be obtained by this process, which is more particularly intended for the 10 production of automobile parts by deep drawing.

Ultrahigh-strength steels have been developed in recent years, especially so as to meet the specific requirements of the automobile industry, which are in 15 particular the reduction in weight, and therefore in thickness, of the parts and the improvement in safety afforded by the increase in fatigue strength and impact behavior of the parts. These improvements must also not degrade the formability of the steel sheet used for 20 producing the parts.

Thus, dual-phase steels have been developed in which the structure is ferritic/martensitic, which make it possible to achieve a tensile strength  $R_m$  of more than 25 400 MPa but which do not have good drawability characteristics, since their mean anisotropy coefficient  $r$  is close to 1. Moreover, their galvanizability is poor, since they contain large amounts of silicon or other elements deleterious to 30 good wetting of the surface of the strip by the molten zinc.

Also known are steels with a single-phase structure, which have a high mean anisotropy coefficient  $r$  but 35 have only moderate mechanical properties, with a tensile strength  $R_m$  not exceeding 400 MPa.

As examples, mention may be made of low-interstitial

steels and aluminum-killed reparkerized steels. Attempts at enhancing the conventional hardening mechanisms for these types of steel fail to appreciably improve their mechanical properties. Furthermore, this 5 steel must be capable of being galvanized.

The object of the present invention is to remedy the drawbacks of the steels of the prior art by proposing a steel strip capable of deep drawing and having at the 10 same time excellent mechanical properties and excellent anisotropy characteristics.

For this purpose, the first subject of the invention is a process for producing a cold-rolled 15 ferritic/martensitic dual-phase steel strip, characterized in that a slab, the chemical composition of which comprises, by weight:

$$0.010\% \leq C \leq 0.100\%$$

$$0.050\% \leq Mn \leq 1.0\%$$

20                     $0.010\% \leq Cr \leq 1.0\%$

$$0.010\% \leq Si \leq 0.50\%$$

$$0.001\% \leq P \leq 0.20\%$$

$$0.010\% \leq Al \leq 0.10\%$$

$$N \leq 0.010\%$$

25 the balance being iron and impurities resulting from the smelting, is hot rolled,

said process then comprising the steps consisting in:

          - coiling the hot-rolled strip obtained at a temperature of between 550 and 850°C; then

30            - cold rolling the strip with a reduction ratio of between 60 and 90%; then

          - annealing the strip continuously in the intercritical range; and

35            - cooling it down to the ambient temperature in one or more steps, the cooling rate between 600°C and the ambient temperature being between 100°C/s and 1500°C/s; and

          - optionally tempering it at a temperature below 300°C,

the annealing and cooling operations being carried out in such a way that the strip finally contains from 1 to 15% martensite.

5 In a preferred method of implementation, the chemical composition of the steel furthermore comprises, by weight:

$$0.020\% \leq C \leq 0.060\%$$

$$0.300\% \leq Mn \leq 0.500\%$$

10  $0.010\% \leq Cr \leq 1.0\%$

$$0.010\% \leq Si \leq 0.50\%$$

$$0.010\% \leq P \leq 0.100\%$$

$$0.010\% \leq Al \leq 0.10\%$$

$$N \leq 0.010\%$$

15 the balance being iron and impurities resulting from the smelting.

The process according to the invention may also include the following features, by themselves or in 20 combination:

- the strip is hot rolled at a temperature above 850°C;

- the strip is hot coiled at a temperature of between 550 and 750°C;

25 - the strip is cold rolled with a reduction ratio of between 70 and 80%;

- the continuous annealing of the cold-rolled strip comprises a temperature rise phase followed by a soak phase at a predetermined temperature;

30 - the soak temperature is between  $Ac_1$  and 900°C;

- the soak temperature is between 750 and 850°C;

- the cooling down to the ambient temperature comprises a first, slow cooling step between the soak temperature and 600°C, during which the cooling rate is

35 less than 50°C/s, followed by a second cooling step at a higher rate, of between 100°C/s and 1500°C/s, down to the ambient temperature.

The second subject of the invention is a cold-rolled

ferritic/martensitic dual-phase steel strip, the chemical composition of which comprises, by weight:

5 In a preferred embodiment, the composition of the strip is the following:

0.020%  $\leq$  C  $\leq$  0.060%  
0.300%  $\leq$  Mn  $\leq$  0.500%  
0.010%  $\leq$  Cr  $\leq$  1.0%  
0.010%  $\leq$  Si  $\leq$  0.50%  
10 0.010%  $\leq$  P  $\leq$  0.100%  
0.010%  $\leq$  Al  $\leq$  0.10%  
N  $\leq$  0.010%

the balance being iron and impurities resulting from the smelting.

15 The steel according to the invention may also include the following features, by themselves or in combination:

- it has a tensile strength  $R_m$  of greater than 20 450 MPa;  
- it has a tensile strength  $R_m$  of greater than 500 MPa;  
- it has a tensile strength  $R_m$  of greater than 600 MPa;  
25 - it has a mean anisotropy coefficient  $r$  of greater than 1.1;  
- it has a mean anisotropy coefficient  $r$  of greater than 1.3;  
- it furthermore contains between 1% and 10% 30 martensite;  
- it furthermore contains between 5% and 8% martensite.

Finally, the third subject of the invention is a steel strip according to the invention for the production of 35 automobile parts by deep drawing.

The process according to the invention consists in hot rolling a slab of specific composition and then in

coiling the hot-rolled strip obtained at a temperature of between 550 and 850°C.

This high-temperature coiling operation is favorable to  
5 the development of what is called a texture, that is to say an anisotropic structure. This is because such a coiling operation makes it possible for the Fe<sub>3</sub>C cementite precipitates to coalesce and to reduce the amount of carbon going back into solution during the  
10 anneal, this being detrimental to the development of the recrystallization texture.

The process then consists in cold rolling the strip with a reduction ratio of between 60 and 90% and then  
15 in annealing the strip continuously in the intercritical range.

The intercritical anneal allows most of the carbide phases formed during the coiling after the  
20 recrystallization to be redissolved. The fact that the austenization and the dissolution of the carbide phases take place after the recrystallization makes it possible to retain the carbon trapped during the recrystallization and to free it once the  
25 recrystallized ferrite texture has developed. The texture will therefore be unaffected by the carbon in solid solution, as is the case with low-temperature coiling, but is only impaired by the isotropic character of the martensite formed.

30 The process then consists in cooling the strip down to the ambient temperature, in one or more steps, the cooling rate between 600°C and the ambient temperature being between 100°C/s and 1500°C/s, and optionally in  
35 tempering it at a temperature below 300°C.

This rapid cooling step allows martensite to form in the structure of the steel, thereby achieving very good mechanical properties. However, measures must be taken

to ensure that too much martensite does not form, as martensite is isotropic and therefore reduces the mean anisotropy coefficient  $r$ .

5 Water quenching allows substantial proportions of carbide phases to be formed in the composition in question. It is possible to reduce the amount of martensitic phase formed by lowering the soak temperature toward lower values in the intercritical 10 range, or else by carrying out a slow cooling operation before the quench.

15 It is also possible to reduce the difference in hardness between the ferritic matrix and the martensitic phase, by cooling the strip more slowly or by performing a short tempering operation, lasting around one minute, on the martensitic phase formed after the water quench.

20 It should be noted that this tempering operation is in no case an overaging treatment, as is found in the prior art. This is because these overaging treatments, which are generally carried out between 300 and 500°C, have in particular the effect of suppressing the 25 martensite, which is an essential element of the present invention. The tempering optionally carried out according to the invention consists in precipitating some of the carbon in solid solution trapped in the martensite, without reducing the proportion of this 30 martensite. The maximum temperature of this tempering operation is 300°C, preferably 250°C and more particularly preferably 200°C.

35 The composition according to the invention includes carbon with a content of between 0.010% and 0.100%. This element is essential for obtaining good mechanical properties but it must not be present in too great an amount, as it would cause an excessive proportion of martensitic phase to be formed.

It also includes manganese with a content of between 0.050% and 1.0%. Manganese improves the yield strength of the steel, but greatly reduces its ductility. This 5 is why its content is limited.

The composition also includes chromium with a content of between 0.010% and 1.0%, which helps in the desired martensite formation.

10 The composition also includes silicon with a content of between 0.010% and 0.50%. This greatly improves the yield strength of the steel, but slightly reduces its ductility and degrades its coatability.

15 The composition also includes phosphorus with a content of between 0.001% and 0.20%, which hardens the microstructure without affecting its texture.

20 The composition also includes aluminum with a content of between 0.010% and 0.10%, which prevents aging by nitrogen trapping.

#### Examples

25 By way of nonlimiting examples, and so as to better illustrate the invention, two grades of steel were produced. Their compositions, in thousandths of a percent, are given in the following table.

	C	Mn	Cr	Si	P	Al	N
A	60	600	70	70	20	56	5
B	43	373	76	13	22	56	5.7

The balance of the compositions consists of iron and inevitable impurities resulting from the smelting.

#### Abbreviations employed

Re: yield strength in MPa;  
Rm: tensile strength in MPa;  
r: anisotropy coefficient;  
P: plateau;  
5 %m: proportion of martensite.

After production, the two grades were austenized at 1250°C for one hour, so as to dissolve the aluminum nitrides. The slabs were then hot rolled in such a way 10 that the end-of-rolling temperature was above 900°C, the value of AR<sub>3</sub> for both grades being about 870°C.

The hot-rolled strips were then cooled by water quenching, at a cooling rate of around 25°C/s, until 15 the coiling temperature was reached. Grade A was coiled at 720°C, while one specimen of grade B was coiled at 550°C and another at 720°C.

The various specimens were then cold rolled so as to 20 achieve a reduction ratio of 75%, then they underwent an annealing treatment at a soak temperature of 750°C in the case of some specimens and 800°C in the case of the others. The cooling down to the ambient temperature was then carried out at a rate of around 25°C/s by 25 water quenching.

Next, the mechanical properties and the anisotropy characteristics of the steels obtained were measured.

30 The results are collated in the following table.

Grade	T <sub>coil</sub> (°C)	T <sub>soak</sub> (°C)	Direction	R <sub>e</sub> (MPa)	R <sub>m</sub> (MPa)	P (%)	r	mean r	%m
A	720	800	T	420	711	0	1.10	0.98	14
			L	405	713	0	1.11		
			45°	425	720	0	0.85		
	750	750	T	443	713	0	1.26	1.02	12
			L	438	717	0	1.13		
			45°	451	736	0	0.84		
B	720	800	T	432	656	0	1.46	1.27	8
			L	430	697	0	1.60		
			45°	436	668	0	1.01		
	750	750	T	454	662	0	2.04	1.37	7
			L	457	690	0	1.41		
			45°	461	677	0	1.01		
	550	800	T	455	677	0	1.47	1.21	6
			L	446	667	0	1.44		
			45°	472	687	0	0.97		
	750	750	T	475	680	0.3	1.46	1.09	5
			L	463	668	0.4	1.25		
			45°	482	697	0.3	0.83		

The overall anisotropy of a steel is determined by the mean normal anisotropy coefficient r:

5

$$r = \frac{r_T + r_L + 2r_{45}}{4}$$

where r<sub>T</sub> denotes the value of r measured in the direction transverse to the rolling direction of the strip, r<sub>L</sub> denotes the value of r measured in the longitudinal or rolling direction of the strip and r<sub>45°</sub> denotes the value of r measured at 45° to the rolling direction of the strip.

15 For a coiling temperature of 720°C, figure 1 shows the relationship between the mean coefficient r and the content of martensite formed %m for grades A and B. It

may be seen that the higher the martensite content, the more anisotropic the steel.

It may also be seen that the higher the martensite  
5 content, the higher the mechanical properties.

As an illustration, figure 2 shows the microstructure  
obtained with grade A, coiled at 720°C and then  
annealed at 750°C in order finally to obtain 12%  
10 martensite. The ferrite and the martensite formed can  
be clearly distinguished in the figure.

CLAIMS

1. A process for producing a cold-rolled  
5 ferritic/martensitic dual-phase steel strip,  
characterized in that a slab, the chemical composition  
of which comprises, by weight:

0.010% ≤ C ≤ 0.100%

0.050% ≤ Mn ≤ 1.0%

10 0.010% ≤ Cr ≤ 1.0%

0.010% ≤ Si ≤ 0.50%

0.001% ≤ P ≤ 0.20%

0.010% ≤ Al ≤ 0.10%

N ≤ 0.010%

15 the balance being iron and impurities resulting from  
the smelting, is hot rolled, said process then  
comprising the steps consisting in:

- coiling the hot-rolled strip obtained at a  
temperature of between 550 and 850°C; then

20 - cold rolling the strip with a reduction ratio of  
between 60 and 90%; then

- annealing the strip continuously in the  
intercritical range; and

25 one or more steps, the cooling rate between 600°C and  
the ambient temperature being between 100°C/s and  
1500°C/s; and

- optionally tempering it at a temperature below  
300°C,

30 the annealing and cooling operations being carried out  
in such a way that the strip finally contains from 1 to  
15% martensite.

2. The process as claimed in claim 1, characterized  
35 in that the chemical composition of the steel  
comprises:

0.020% ≤ C ≤ 0.060%

0.300% ≤ Mn ≤ 0.500%

0.010% ≤ Cr ≤ 1.0%

0.010% ≤ Si ≤ 0.50%

0.010% ≤ P ≤ 0.100%

0.010% ≤ Al ≤ 0.10%

5 N ≤ 0.010%

the balance being iron and impurities resulting from the smelting.

3. The process as claimed in either of claims 1 and 10 2, characterized in that the strip is hot rolled at a temperature above 850°C.

4. The process as claimed in any one of claims 1 to 15 3, characterized in that the strip is hot rolled at a temperature of between 550 and 750°C.

5. The process as claimed in any one of claims 1 to 4, characterized in that the strip is cold rolled with a reduction ratio of between 70 and 80%.

20 6. The process as claimed in any one of claims 1 to 5, characterized in that the continuous annealing of the cold-rolled strip comprises a temperature rise phase followed by a soak phase at a predetermined 25 temperature.

7. The process as claimed in claim 6, characterized in that the soak temperature is between  $Ac_1$  and 900°C.

30 8. The process as claimed in claim 7, characterized in that the soak temperature is between 750 and 850°C.

9. The process as claimed in any one of claims 1 to 35 8, characterized in that the cooling down to the ambient temperature comprises a first, slow cooling step between the soak temperature and 600°C, during which the cooling rate is less than 50°C/s, followed by a second cooling step at a higher rate, of between 100°C/s and 1 500°C/s, down to the ambient temperature.

10. The process as claimed in claim 9, characterized in that the second cooling step is carried out by water quenching.

5

11. The process as claimed in any one of claims 1 to 8, characterized in that the cooling is carried out in a single operation at a cooling rate of between 100°C/s and 1500°C/s.

10

12. The process as claimed in claim 11, characterized in that the cooling is carried out by water quenching.

13. A cold-rolled ferritic/martensitic dual-phase steel strip, the chemical composition of which comprises, by weight:

0.010% ≤ C ≤ 0.100%  
0.050% ≤ Mn ≤ 1.0%  
0.010% ≤ Cr ≤ 1.0%  
0.010% ≤ Si ≤ 0.50%  
0.001% ≤ P ≤ 0.20%  
0.010% ≤ Al ≤ 0.10%

N ≤ 0.010%

the balance being iron and impurities resulting from the smelting, the strip furthermore containing between 1% and 15% martensite.

14. The steel strip as claimed in claim 13, characterized in that its chemical composition furthermore comprises:

0.020% ≤ C ≤ 0.060%  
0.300% ≤ Mn ≤ 0.500%  
0.010% ≤ Cr ≤ 1.0%  
0.010% ≤ Si ≤ 0.50%  
0.010% ≤ P ≤ 0.100%  
0.010% ≤ Al ≤ 0.10%  
N ≤ 0.010%

the balance being iron and impurities resulting from the smelting.

15. The steel strip as claimed in either of claims 13 and 14, characterized in that it has a tensile strength  $R_m$  of greater than 450 MPa.

5

16. The steel strip as claimed in claim 15, characterized in that it has a tensile strength  $R_m$  of greater than 500 MPa.

10 17. The steel strip as claimed in claim 16, further characterized in that it has a tensile strength  $R_m$  of greater than 600 MPa.

15 18. The steel strip as claimed in any one of claims 13 to 17, characterized in that it has a mean anisotropy coefficient  $r$  of greater than 1.1.

19. The steel strip as claimed in claim 18, further characterized in that it has a mean anisotropy coefficient  $r$  of greater than 1.3.

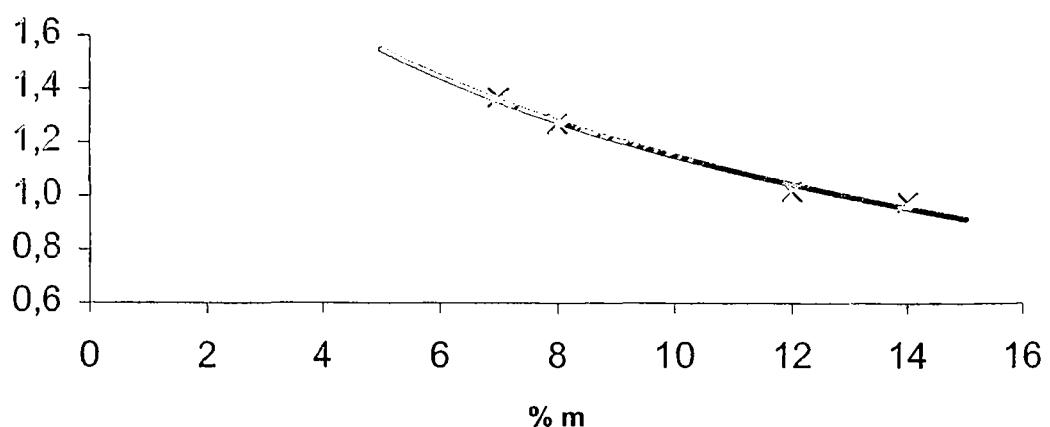
20. The steel strip as claimed in any one of claims 13 to 19, characterized in that it furthermore contains between 1% and 10% martensite.

25

21. The steel strip as claimed in claim 20, characterized in that it furthermore contains between 5% and 8% martensite.

30 22. The use of a steel strip as claimed in any one of claims 13 to 21 for the production of automobile parts by deep drawing.

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Fig. 1Fig. 2