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# (54) METHOD OF PRODUCING AN AUSTENITIC STEEL

VERFAHREN ZUR HERSTELLUNG VON AUSTENITISCHEM STAHL

PROCÉDÉ DE PRODUCTION D'ACIER AUSTÉNITIQUE

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(56) References cited:
**EP-A1- 2 090 668**
**EP-A1- 2 208 803**
**WO-A1-93/13233**
**WO-A1-2006/082104**
**WO-A1-2009/084792**
**WO-A1-2009/095264**
**US-A1- 2008 035 249**

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## Description

**[0001]** The invention relates to a method of producing an austenitic steel sheet excellent in resistance to delayed cracking.

**[0002]** In view of fuel economy and safety in the case of collisions, high strength steels are used more and more in the automobile industry. This requires the use of structural materials that combine a high tensile strength with high ductility. Austenitic alloys comprising as main elements iron, carbon and high levels of manganese, which can be hot-rolled or cold-rolled and have a strength that may exceed 1000 MPa. The mode of deformation of these steels depends on the stacking fault energy: for a sufficiently high stacking fault energy, the observed mode of mechanical deformation is twinning, which results in a high work hardenability. By acting as an obstacle to the propagation of dislocations, the twins increase the flow stress. However, when the stacking fault energy exceeds a certain limit, slip of perfect dislocations becomes the main deformation mechanism and the work hardening is reduced. It is known that the sensitivity to delayed cracking increases with the mechanical strength, in particular after certain cold-forming operations since high residual tensile stresses are liable to remain after deformation. In combination with atomic hydrogen possibly present in the metal, these stresses are liable to result in delayed cracking, that is to say cracking that occurs a certain time after the deformation itself. Hydrogen may progressively build up by diffusion to crystal lattice defects, such as matrix/inclusion interfaces, twin boundaries and grain boundaries. It is in the latter areas that hydrogen may become harmful when it reaches a critical concentration after a certain time. For a constant grain size, the time required to attain a critical level depends on the initial concentration of mobile hydrogen, the intensity of the residual stress concentration field and the kinetics of hydrogen diffusion.

**[0003]** In particular circumstances, small amounts of hydrogen may be introduced at some stages of steel fabrication such as chemical or electrochemical pickling, annealing under special atmospheres, electroplating or hot dip galvanizing. Subsequent machining operations using lubricating oils and greases may also cause hydrogen production after decomposition of these substances at high temperatures.

**[0004]** It is an object of this invention to provide a method of producing an austenitic steel sheet excellent in resistance to delayed cracking.

**[0005]** It is a further object of this invention to provide a method of producing an austenitic steel sheet which is energy efficient and simple in comparison to the conventional route for this type of steel.

**[0006]** According to the invention one or more of these objects is reached by providing a method of producing an austenitic steel sheet excellent in resistance to delayed cracking comprising

- casting an ingot, or a continuously cast slab, or a continuously cast thin slab or a strip-cast strip, the composition thereof comprising, in weight:

  - 0.50% - 0.80 %C
  - 10 - 17% Mn
  - 1.0 - 5% Al
  - at most 0.5% Si
  - at most 0.020% S
  - at most 0.050% P
  - 50 - 200 ppm N
  - 0.050 - 0.25 % V

  the remainder being iron and unavoidable impurities inherent to fabrication;

- providing a hot-rolled strip by hot rolling the ingot, the continuously cast slab, the continuously cast thin slab or the strip-cast strip to the desired hot rolled thickness.

- cold-rolling the hot-rolled strip to the desired final thickness,

- continuous annealing of the cold-rolled strip in a process comprising heating up the strip at a heating rate $V_h$ of between 3 and 60°C/s to an annealing temperature $T_a$ for an annealing time $t_a$ of between 15 and 300 seconds followed by cooling at a cooling rate $V_c$ of between 10 and 100°C/s and wherein $T_a$ is from 750 to 850°C.

**[0007]** By using a high aluminium content the SFE of the steel increases. Any adverse effects of the elements that lower the SFE, such as silicon, are counteracted by the addition of aluminium. Additionally, aluminium lowers the activity and the diffusivity of carbon in austenite, which reduces the driving force for forming carbides. The vanadium, which is added as an essential alloying addition, forms carbides. These vanadium-carbides act as hydrogen sinks if and when

the size and distribution of the vanadium-carbides is correct. The increased aluminium content is therefore essential to control the vanadium-carbide precipitation because it prevents the vanadium-carbides from coarsening due to the reduced carbon activity and diffusivity as a result of the presence of aluminium. The inventors found that at least 1.0 % Al and from 0.050 to 0.25 % V is required to achieve this. Lower aluminium contents led to too coarse vanadium-carbides, thereby rendering them ineffective as hydrogen sinks, and the amount of Vanadium needs to be controlled between the mentioned values to achieve a sufficient number of small precipitates. Higher V-values lead to early nucleation of the precipitates and thereby inevitably to coarse and fewer precipitates, whereas values below 0.050% V simply result in to few precipitates, even if they are fine enough. The annealing treatment is crucial in that it controls the precipitation of the vanadium-carbides and causes the recrystallisation of the cold-deformed microstructure caused by the cold-rolling to result in a fine grain structure. In a preferable embodiment the silicon content is very low, i.e. at impurity level. In principle the aluminium content is limited only by the fact that the steel according to the invention is an austenitic steel.

**[0008]** EP2090668-A1 discloses a method of producing high strength in a Cr-bearing TWIP steel by subjecting the steel to a cold reduction. US20080035249-A1 disclose TWIP steels with Mn contents of over 21%.

**[0009]** The maximum aluminium content is 5%. Preferably the aluminium content is at least 1.25 and/or at most 3.5%, more preferably at least 1.5 and/or at most 2.5%.

**[0010]** In an embodiment the maximum annealing temperature $T_a$ is 825°C or even 800°C. Preferably the cooling rate $V_c$ is between 20 and 80 °C/s. The heating rate is according to the invention is between 3 and 60°C/s. The annealing time $t_a$ according to the invention is between 15 and 300 seconds.

**[0011]** In a preferable embodiment, the maximum annealing temperature $T_a$ is from 775 to 795 °C (i.e. 785 $\pm$ 10°C).

**[0012]** Preferably, the steel strip material has been pickled before cold rolling. Pickling is (often) necessary before cold rolling to remove oxides, to prevent rolling in of oxides. Preferably, the cold rolled strip material is produced from a hot rolled strip material or a belt cast strip material.

**[0013]** In a preferred embodiment of the invention during the cooling at a cooling rate $V_c$ after the continuous annealing the strip is led through a hot dip bath for providing a metallic coating by hot dipping the strip into a molten bath of the metal making up the metallic coating. This process leads to a very economical and quick process to produce a metallically coated steel strip. The metallic coating can be any known common coating such as zinc or zinc alloy, wherein the zinc may be alloyed with elements such as aluminium and/or magnesium.

**[0014]** In another embodiment of the invention the strip is pickled after continuous annealing and wherein the strip is provided with a metallic coating by pickling after annealing followed by heating to a temperature below the continuous annealing temperature before the strip is led through a hot dip bath for providing a metallic coating by hot dipping the strip into a molten bath of the metal making up the metallic coating. This alternative process is available if the economical process as described hereinabove is not preferred. There may be issues with adhesion with certain specific metallic coatings for which a pickling treatment may be necessary. After pickling, it is not necessary nor desirable to heat the strip above the $T_a$. It is preferable that the heating temperature remain below $T_a$.

**[0015]** With this method, the strip material is heated only to a temperature high enough to form a closed inhibition layer. This temperature is lower than the normal continuous annealing temperature necessary for metallurgical reasons (such as recrystallisation to influence mechanical properties). The forming of oxides on the surface of the steel strip material is thereby reduced.

**[0016]** Preferably, the temperature below the continuous annealing temperature is between 400 and 600 °C. In this temperature range the forming of oxides is considerably reduced and the strip material is heated sufficiently for the subsequent hot dip galvanizing.

**[0017]** According to a preferred embodiment, the Fe in the strip material is reduced during or after the heating to a temperature below the continuous annealing temperature and before the hot dip galvanising. By reducing the strip material, the Fe-oxides that are formed are reduced, and in this way the amount of oxides present on the surface of the strip material before hot dip galvanizing is decreased considerably.

**[0018]** Preferably, the reduction is performed using $H_2N_2$, more preferably using 5 - 30% $H_2N_2$ in the reducing atmosphere. It has been found that with the use of this atmosphere most oxides can be removed.

**[0019]** According to a preferred embodiment, an excess amount of $O_2$ is provided in the atmosphere during or after the heating of the strip material and before the reduction of the strip material. The providing of an excess amount of oxygen improves the quality of the surface of the steel strip material before the hot dip galvanizing, and thus the quality of the zinc layer coated on the AHSS strip material. It is supposed that the oxygen binds the alloying elements in the AHSS strip material both at the surface of the strip material and internally, and that in this way the oxides formed cannot migrate to the surface of the strip material.

**[0020]** The reducing atmosphere that follows after the oxidation will then reduce the oxides at the surface of the strip material, and in this way the amount of oxides at the surface of the strip material is considerably reduced or even almost absent, as experiments have shown. Preferably, the excess amount of $O_2$ is provided in an amount of 0,05 - 5 % $O_2$. This amount of oxygen has been found to suffice.

**[0021]** In a preferred embodiment of the invention V-alloyed TWIP steel strip material according to the invention was

hot rolled, pickled and cold rolled, and continuously annealed to a temperature according to the invention and pickled again. Then the strip material is heated to a temperature of 527 °C in an annealing line, and thereafter hot dip galvanised in a galvanising bath at approximately 450°C.

**[0022]** During the heating of the strip material to the temperature of 527 °C, an excess amount of 1% 02 is provided. The oxygen is provided at such a high temperature so that not only forms oxides at the surface of the strip material, but also at some depth under the surface binds the alloying elements. After the providing of the oxygen, the strip material is reduced using approximately 5% $H_2N_2$. The reduction of the strip material removes the oxides from the surface, but the oxides formed under the surface remain where they are and cannot migrate to the surface.

**[0023]** Thus, by reducing the surface the oxides are effectively removed and no new oxides can be formed at the surface. These oxides, when not removed, cause a bad adhesion of the zinc layer to the substrate, resulting in bare spots, flaking and the forming of cracks in the zinc layer when the material is bent. It is presumed that by normal reduction, the alloying elements migrate to the surface very fast at the alloying temperature and thus form oxides at the surface again before the hot dip galvanising takes place. Whatever the exact mechanism may be, it has been found that the use of this method diminishes or almost eliminates the amount of oxides found in a hot dip galvanised zinc layer on a V-alloyed TWIP steel.

**[0024]** In an embodiment of the invention the cold-rolling reduction is between 10 to 90%, more preferably between 30 and 85, even more preferably between 45 and 80%.

**[0025]** In an embodiment of the invention the annealed strip is temper rolled with a reduction of from 0.5 to 10% prior to or after the metallic coating has been provided to the strip.

**[0026]** In an embodiment of the invention the Vanadium content is between 0.06 and 0.22%.

**[0027]** In a second aspect of the invention a strip or sheet is provided produced by a method according to any one of claims 1 to 6, wherein the steel preferably is provided with a metallic coating. In a preferred embodiment of the invention the strip or sheet is used for the production of automotive inner or outer parts or wheels or for hydroforming applications.

**[0028]** The invention will now be further explained by means of the following, non-limitative examples:
The chemical compositions of the materials used in this study are shown in Table 1.

Table 1 Chemical composition of the materials studied (incl. typical semi-industrial Si-free reference material) (all in wt%, Balance: Fe and impurities)

| Code | C | Si | Mn | Al | V | S | N | P |
|---|---|---|---|---|---|---|---|---|
| 8V-1 | 0.74 | 0.36 | 13.8 | 2.2 | 0.080 | 0.011 | 0.011 | Low* |
| 8V-2 | 0.74 | 0.36 | 13.8 | 2.3 | 0.086 | 0.011 | 0.009 | Low |
| 8V-3 | 0.74 | 0.31 | 13.7 | 2.3 | 0.084 | 0.011 | 0.007 | Low |
| 10V | 0.71 | 0.22 | 13.4 | 2.4 | 0.106 | 0.010 | 0.011 | Low |
| 16V | 0.72 | 0.25 | 14.5 | 2.3 | 0.160 | 0.001 | 0.006 | 0.008 |
| 21V | 0.69 | 0.21 | 14.9 | 2.6 | 0.213 | 0.010 | - | 0.005 |
| Si-free | 0.70 | <0.20 | 14.5 | 2.5 | impurity | 0.004 | 0.007 | 0.030 |
| * Low = impurity level | | | | | | | | |

**[0029]** The finish rolling temperature (FRT) was chosen to ensure recrystallisation of the deformed microstructure and the coiling temperature was kept below 500°C to avoid carbide precipitation. Recrystallisation does not solely depend on the FRT but also depends on the time, on the rolling strain accumulated since the last recrystallisation event during hot rolling and on the strain rate.

**[0030]** All hot rolled materials were 50% cold rolled and subsequently recrystallisation annealed. Different annealing cycles were applied to determine the optimal annealing parameters. Note that the elongations were between 45% and 50% for all samples, except for those that were not recrystallised (36-45%) and the material annealed 920°C (65%). Since the strength is considered more important, the following discussion will focus on that.

**[0031]** For annealing temperatures up to 750°C, the material softens due to an increased fraction of recrystallised material and probably some grain growth. At these temperatures, the effect of precipitation is limited. The difference between the (fully recrystallised) materials annealed at 775°C and 800°C is small because the precipitation is considered optimal in this temperature region for minimising grain growth. Based on these observations, the recommended annealing temperature is 785°C $\pm$ 10°C.

**Table 1 Mechanical properties of the materials after 50% cold rolling and annealing.**

| Grade Si-free | Heating rate (°C/s) | $T_a$ (°C) | $t_a$ (s) | Rp (MPa) | Rm (MPa) | Ag (%) | Atot (%) |
|---|---|---|---|---|---|---|---|
| Si-free | 5 | 700 | 270 | 580 | 920 | | 43 |
| Si-free | 5 | 750 | 270 | 546 | 900 | | 46 |
| Si-free | 5 | 800 | 270 | 490 | 875 | | 47 |
| Si-free | 5 | 850 | 270 | 450 | 850 | | 48 |
| Si-free | 5 | 920 | 270 | 410 | 870 | | 65 |
| 8V-1 | 5 | 785 | 270 | 584 | 962 | 44 | 46 |
| 8V-2 | 5 | 785 | 270 | 595 | 970 | 45 | 49 |
| 8V-3 | 5 | 785 | 270 | 585 | 964 | 46 | 50 |
| 10V | 5 | 785 | 270 | 575 | 939 | 45 | 49 |
| 16V | 5 | 700 | 270 | 764 | 1072 | 32 | 36 |
| 16V | 5 | 725 | 270 | 687 | 1012 | 37 | 41 |
| 16V | 5 | 750 | 270 | 636 | 977 | 41 | 45 |
| 16V | 5 | 775 | 270 | 614 | 964 | 42 | 46 |
| 16V | 5 | 800 | 270 | 590 | 946 | 43 | 48 |
| 16V | 5 | 750 | 60 | 670 | 1004 | 39 | 43 |
| 16V | 5 | 775 | 60 | 626 | 973 | 41 | 45 |
| 16V | 5 | 800 | 60 | 597 | 955 | 44 | 48 |
| 21V | 30 | 700 | 60 | 655 | 985 | | 45 |
| 21V | 30 | 750 | 60 | 600 | 950 | | 43 |
| 21V | 30 | 920 | 60 | 425 | 835 | | 53 |

**[0032]** The delayed cracking and stress corrosion cracking results on the V-alloyed grades show a lower susceptibility to crack formation as the material is annealed at a higher temperature. For the stress corrosion cracking sensitivity, V addition is clearly beneficial at an annealing temperature of 750°C, but also at higher annealing temperatures.

**[0033]** The V-alloys were subjected to a resistance spot welding tests. Hot cracking in the weld was largely reduced compared to the Si-free non V-alloyed material.

## Claims

**1.** A method of producing an austenitic steel sheet excellent in resistance to delayed cracking, comprising

- casting an ingot, or a continuously cast slab, or a continuously cast thin slab or a strip-cast strip, the composition thereof comprising, in weight:

  - 0.50% - 0.80 %C
  - 10 - 17% Mn
  - 1.0 - 5% Al
  - at most 0.5% Si
  - at most 0.020% S
  - at most 0.050% P
  - 50 - 200 ppm N
  - 0.050 - 0.25 % V

  the remainder being iron and unavoidable impurities inherent to fabrication;
- providing a hot-rolled strip by hot rolling the ingot, the continuously cast slab, the continuously cast thin slab

or the strip-cast strip to the desired hot rolled thickness.
- cold-rolling the hot-rolled strip to the desired final thickness,
- continuous annealing of the cold-rolled strip in a process comprising heating up the strip at a heating rate $V_h$ of between 3 and 60°C/s to an annealing temperature $T_a$ for an annealing time $t_a$ of between 15 and 300 seconds followed by cooling at a cooling rate $V_c$ of between 10 and 100°C/s and wherein $T_a$ is from 750 to 850°C.

2. Process according to claim 1 wherein the aluminium content is at least 1.25 and/or at most 3.5%.

3. Process according to claim 1 wherein during the cooling at a cooling rate $V_c$ after the continuous annealing the strip is led through a hot dip bath for providing a metallic coating by hot dipping the strip into a molten bath of the metal making up the metallic coating.

4. Process according to claim 1 wherein the strip is pickled after continuous annealing and wherein the strip is provided with a metallic coating by pickling after annealing followed by heating to a temperature below the continuous annealing temperature before the strip is led through a hot dip bath for providing a metallic coating by hot dipping the strip into a molten bath of the metal making up the metallic coating.

5. Process according to any one of the preceding claims wherein the cold-rolling reduction is between 10 to 90%, more preferably between 30 and 85, even more preferably between 45 and 80%.

6. Process according to any one of the preceding claims wherein the annealed strip is temper rolled with a reduction of from 0.5 to 10% prior to or after the metallic coating has been provided to the strip.

7. Process according to any one of the preceding claims wherein the Vanadium content is between 0.06 and 0.22%.

8. Process according to any one of the preceding claims wherein the cooling rate $V_c$ is between 20 and 80°C/s.

9. Process according to any one of the claims 1, 2 or 4 to 8 wherein the strip is pickled after continuous annealing and wherein the strip is provided with a metallic coating, by pickling after continuous annealing followed by heating to a temperature between 400 and 600 °C before the strip is led through a hot dip bath for providing a metallic coating by hot dipping the strip into a molten bath of the metal making up the metallic coating.

10. Process according to claim 9 wherein the Fe in the strip material is reduced during or after the heating to a temperature below the continuous annealing temperature and before the hot dip galvanising, preferably wherein the reduction is performed using $H_2N_2$, more preferably using 5 - 30% $H_2N_2$ in the reducing atmosphere.

11. Process according to claim 10 wherein an excess amount of $O_2$ is provided in the atmosphere during or after the heating of the strip material and before the reduction of the strip material, preferably wherein the excess amount of $O_2$ is provided in an amount of 0,05 - 5 % $O_2$.

12. Process according to any one of the preceding claims wherein the calling temperature after hot rolling is kept below 500°C and/or wherein the annealing temperature $T_a$ is 785°C $\pm$ 10°C.

13. Strip or sheet produced by a method according to any one of claims 1 to 12, wherein the steel preferably is provided with a metallic coating.

14. Use of a strip or sheet according to claim 13 for the production of automotive inner or outer parts or wheels.

15. Use of a strip or sheet according to claim 13 for hydroforming applications.

**Patentansprüche**

1. Verfahren zur Herstellung eines austenitischen Stahlblechs mit ausgezeichnete Widerstandsfähigkeit gegen verzögerte Rissbildung, umfassend:

- Gießen eines Blocks oder einer stranggegossenen Bramme oder einer stranggegossenen Dünnbramme oder eines bandgegossenen Bands, wobei die Zusammensetzung davon in Gewichtsprozent folgendes umfasst:

- 0,50 bis 0,80 % C,
- 10 bis 17% Mn,
- 1,0 bis 5% Al,
- höchstens 0,5% Si,
- höchstens 0,020% S,
- höchstens 0,050% P,
- 50 bis 200 ppm N,
- 0,050 bis 0,25% V,

wobei der Rest Eisen und unvermeidbare Unreinheiten ist, die bei der Fertigung inhärent sind;
- Bereitstellen eines heißgewalzten Bands durch Heizwalzen des Blocks, der stranggegossenen Bramme, der stranggegossenen Dünnbramme oder des bandgegossenen Bands auf die gewünschte heißgewalzte Dicke;
- Kaltwalzen des heißgewalzten Bands auf die gewünschte finale Dicke;
- Durchlaufglühen des kaltgewalzten Bands in einem Prozess, der das Erhitzen des Bands mit einer Erhitzungsrate $V_h$ zwischen 3 und 60 °C/s auf eine Glühtemperatur $T_a$ über eine Glühzeit $t_a$ zwischen 15 und 300 Sekunden umfasst, gefolgt vom Kühlen mit einer Kühlrate $V_c$ zwischen 10 und 100 °C/s, und wobei Ta zwischen 750 und 850 °C liegt.

**2.** Verfahren nach Anspruch 1, wobei der Aluminiumanteil wenigstens 1,25 und/oder höchstens 3,5% beträgt.

**3.** Verfahren nach Anspruch 1, wobei während dem Kühlen mit einer Kühlrate $V_c$ das Band nach dem Durchlaufglühen durch ein Feuerverzinkungsbad geführt wird, um eine metallische Beschichtung durch Feuerverzinken des Bands in einem Schmelzbad des die metallische Beschichtung bildenden Metalls bereitzustellen.

**4.** Verfahren nach Anspruch 1, wobei das Band nach dem Durchlaufglühen gebeizt wird, und wobei das Band mit einer metallischen Beschichtung durch Beizen nach dem Glühen versehen wird, gefolgt von einer Erhitzung auf eine Temperatur unterhalb der Temperatur des Durchlaufglühens, bevor das Band durch ein Feuerverzinkungsbad geführt wird, um eine metallische Beschichtung durch Feuerverzinken des Bands in einem Schmelzbad des die metallische Beschichtung bildenden Metalls bereitzustellen.

**5.** Verfahren nach einem der vorstehenden Ansprüche, wobei die Reduzierung durch Kaltwalzen zwischen 10 und 90% liegt, vorzugsweise zwischen 30 und 85%, mehr bevorzugt zwischen 45 und 80%.

**6.** Verfahren nach einem der vorstehenden Ansprüche, wobei das geglühte Band mit einer Reduzierung zwischen 0,5 und 10% nachgewalzt wird bevor oder nachdem die metallische Beschichtung an dem Band bereitgestellt wird bzw. worden ist.

**7.** Verfahren nach einem der vorstehenden Ansprüche, wobei der Vanadiumgehalt zwischen 0,06 und 0,22% liegt.

**8.** Verfahren nach einem der vorstehenden Ansprüche, wobei die Kühlrate $V_c$ zwischen 20 und 80 °C/s liegt.

**9.** Verfahren nach einem der Ansprüche 1, 2 oder 4 bis 8, wobei das Band nach dem Durchlaufglühen gebeizt wird, und wobei das Band mit einer metallischen Beschichtung versehen wird durch Beizen nach dem Durchlaufglühen, gefolgt von Erhitzen auf eine Temperatur zwischen 400 und 600 °C, bevor das Band durch ein Feuerverzinkungsbad geführt wird, um eine metallische Beschichtung durch Feuerverzinken des Bands in einem Schmelzbad des die metallische Beschichtung bildenden Metalls bereitzustellen.

**10.** Verfahren nach Anspruch 9, wobei das Fe in dem Bandmaterial während oder nach dem Erhitzen auf eine Temperatur unterhalb der Temperatur des Durchlaufglühens und vor der Feuerverzinkung reduziert wird, wobei die Reduzierung vorzugsweise unter Verwendung von $H_2N_2$, noch bevorzugter von 5 bis 30% $H_2N_2$ in der Reduzierungsatmosphäre durchgeführt wird.

**11.** Verfahren nach Anspruch 10, wobei eine überschüssige Menge $O_2$ in der Atmosphäre bereitgestellt wird während oder nach dem Erhitzen des Bandmaterials und vor der Reduzierung des Bandmaterials, wobei die überschüssige Menge $O_2$ vorzugsweise in einer Menge von 0,05 bis 5% $O_2$ bereitgestellt wird.

**12.** Verfahren nach einem der vorstehenden Ansprüche, wobei die Kühltemperatur nach dem Heißwalzen unter 500 °C gehalten wird, und/oder wobei die Glühtemperatur $T_a$ 785 °C $\pm$ 10 °C beträgt.

13. Band oder Blech, hergestellt durch ein Verfahren nach einem der Ansprüche 1 bis 12, wobei der Stahl vorzugsweise mit einer metallischen Beschichtung bereitgestellt ist.

14. Verwendung eines Bands oder Blechs nach Anspruch 13 zur Herstellung von inneren oder äußeren Bauteilen von Kraftfahrzeugen oder Rädern.

15. Verwendung eines Bands oder Blechs nach Anspruch 13 für Hydroformanwendungen.

**Revendications**

1. Procédé de production d'une feuille en acier austénitique présentant une excellente résistance à la fissuration retardée, comprenant les étapes consistant à couler un lingot, ou une dalle coulée en continu, ou une mince dalle coulée en continu ou une bande coulée en bande mince, dont la composition comprend, en poids

   de 0,50 % à 0,80 % de C
   de 10 à 17% de Mn
   de 1,0 à 5% d'Al
   au plus 0,5 % de Si
   au plus 0,020 % de S
   au plus 0,050 % de P
   de 50 à 200 ppm de N
   de 0,050 à 0,25 % de V

   le reste étant du fer et des impuretés inévitables inhérentes à la fabrication;
   fournir une bande laminée à chaud en laminant à chaud le lingot, la dalle coulée en continu ou la bande coulée en bande mince à l'épaisseur laminée à chaud souhaitée,
   laminer à froid la bande laminée à chaud à l'épaisseur finale souhaitée,
   recuire en continu la bande laminée à froid dans un processus comprenant le chauffage de la bande à une vitesse de chauffage $V_h$ comprise entre 3 et 60 °C/s à une température de recuit $T_a$ pendant un temps de recuit $t_a$ compris entre 15 et 300 secondes suivi par le refroidissement à une vitesse de refroidissement $V_c$ comprise entre 10 et 100 °C/s et $t_a$ étant compris entre 750 et 850 °C.

2. Procédé selon la revendication 1, la teneur en aluminium étant d'au moins 1,25 et/ou au plus de 3,5 %.

3. Procédé selon la revendication 1, au cours du refroidissement à une vitesse de refroidissement $V_c$ après le recuit continu, la bande étant dirigée à travers bain d'immersion chaud pour appliquer un revêtement métallique par immersion à chaud de la bande dans un bain fondu du métal constituant le revêtement métallique.

4. Procédé selon la revendication 1, la bande étant décapée après recuit continu et la bande étant pourvue d'un revêtement métallique par décapage après recuit suivi par chauffage à une température inférieure à la température de recuit continu avant que la bande ne soit amenée à un bain d'immersion chaud pour appliquer un revêtement métallique par immersion à chaud de la bande dans un bain fondu du métal constituant le revêtement métallique.

5. Procédé selon l'une quelconque des revendications précédentes, la réduction par laminage à froid étant comprise entre 10 et 90 %, encore de préférence entre 30 et 85, encore plus de préférence entre 45 et 80 %.

6. Procédé selon l'une quelconque des revendications précédentes, la bande recuite étant écrouie à froid avec une réduction de 0,5 à 10 % avant ou après que le revêtement métallique a été appliqué sur la bande.

7. Procédé selon l'une quelconque des revendications précédentes, la teneur en vanadium étant comprise entre 0,06 et 0,22 %.

8. Procédé selon l'une quelconque des revendications précédentes, la vitesse de refroidissement $V_c$ étant comprise entre 20 et 80 °C/s.

9. Procédé selon l'une quelconque des revendications 1, 2 ou 4 à 8, la bande étant décapée après recuit continu et la bande étant pourvue d'un revêtement métallique par décapage après recuit suivi par chauffage à une température

comprise entre 400 et 600 °C avant que la bande ne soit amenée à un bain d'immersion chaud pour appliquer un revêtement métallique par immersion à chaud de la bande dans un bain fondu du métal constituant le revêtement métallique.

**10.** Procédé selon la revendication 9, le Fe dans le matériau de bande étant réduit pendant ou après le chauffage à une température inférieure à la température de recuit continu et avant la galvanisation par immersion à chaud, de préférence, la réduction étant effectuée à l'aide de $H_2N_2$, de préférence à l'aide de 5 à 30 % de $H_2N_2$ dans l'atmosphère réductrice.

**11.** Procédé selon la revendication 10, une quantité excédentaire de $O_2$ étant fournie dans l'atmosphère pendant ou après le chauffage du matériau de bande et avant la réduction du matériau de bande, de préférence la quantité excédentaire de $O_2$ étant fournie dans une quantité comprise entre 0,05 et 5 % de $O_2$.

**12.** Procédé selon l'une quelconque des revendications précédentes, la température de refroidissement après le laminage à chaud étant maintenue en dessous de 500 °C et/ou la température de recuit $T_a$ étant de 785 °C $\pm$10 °C.

**13.** Bande ou feuille produite par un procédé selon l'une quelconque des revendications 1 à 12, l'acier étant de préférence pourvu d'un revêtement métallique.

**14.** Utilisation d'une bande ou feuille selon la revendication 13 pour la production de roues ou pièces internes ou externes pour automobile.

**15.** Utilisation d'une bande ou feuille selon la revendication 13 pour des applications d'hydroformage.

## REFERENCES CITED IN THE DESCRIPTION

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### Patent documents cited in the description


- EP 2090668 A1 **[0008]**
- US 20080035249 A1 **[0008]**