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(54) Low-carbon precipitation-strengthened steel for cold heading applications

(57) A rolled steel wire or rod suitable for the production of cold headed products without the application of heat treating operations has a chemical composition, by weight, of:
$0.04\% \le C \le 0.1\%$
$1.8\% \le Mn \le 2.0\%$
$0.15\% \le Si \le 0.30\%$
$S \le 0.025\%$
$P \le 0.025\%$
$Cr \le 0.50\%$
$Mo \le 0.08\%$
$Ni+Cu \ge 0.30\%$
$0.01\% \le Al \le 0.05\%$
$V \le 0.05\%$
$10 \text{ ppm} \le B \le 30 \text{ ppm}$
$N \le 100 \text{ ppm}$
$Ti \ge 0.06\%$

the remainder being iron and impurities resulting from the production process. The steel has a cementite-free microstructure comprising a predominant phase, a minor phase and MX-precipitations distributed within the predominant phase, the predominant phase consisting of bainitic ferrite, the minor phase comprising retained austenite and optionally martensite with a size of 2 to 3 μm, the relative amount of the minor phase amounting to < 20% by volume.

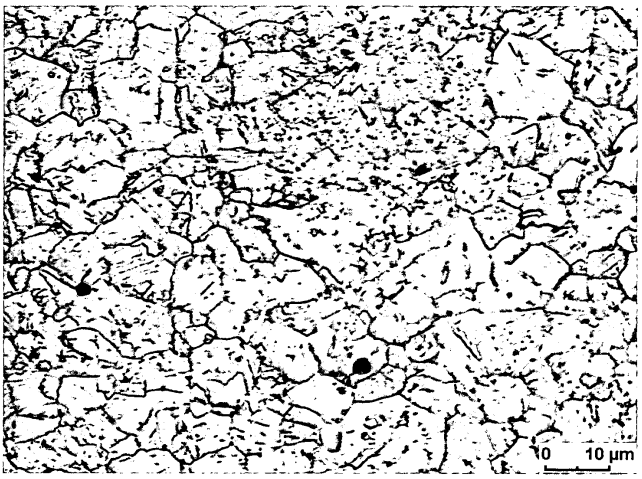


Fig. 1

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Description

Field of the Invention

[0001] The present invention relates to low-carbon precipitation-strengthened bainitic steels with predomnantly granular bainite microstructure in the wire and rod that are suitable for the production of cold headed products without the application of heat treating operations.

Background of the Invention

[0002] Conventionally, two thermal treatments are applied during the production of fasteners and machinery elements, namely, (i) softening annealing prior to cold deformation and (ii) heat treatment after cold deformation to obtain the required property specifications. Quite recently, the development of a dual-phase steel (Dupla™) by Corus enabled the production of high strength fasteners without the need for heat treatments. New steel grades for fasteners manufacturing are required to have good workability in the cold heading operations and to exhibit a sufficient strength level prior to shaping along with the appropriate strain hardening characteristics to develop the required properties in the ultimate product. This combination of strength and workability is possible in the dual-phase steel due to the predominantly ferritic microstructure with a volume fraction of martensite, bainite and retained austenite in the range of 10 to 20%.

[0003] Regarding the use of bainitic steels for cold heading applications, some low and ultra low carbon steels have been considered as good candidates for the production of cold forged parts and fasteners that would allow the achievement of required properties of the final product without heat treatment. Their mechanical properties are characterised by relatively low yield strength and appropriate strain hardening characteristics capable of developing high strength properties after drawing and forging stages of fasteners production process. Bainitic steels for cold forming of products have been disclosed in several patents. For example, there are descriptions in the following patents: Ascometal (EP0851038A1), Mittal Grandrange (EP1565587A1), Saarstahl (EP1780293A2), Mittal Grandrange (FR 2867785). However, the main concept of all these steels lies in the proper control of carbon and alloying elements content to achieve the required mechanical properties.

[0004] The strength of these known steels is derived from the following mechanisms:

- Solid solution strengthening
- Structural strengthening
- Dislocation strengthening
- Second (hard) phase strengthening
- Strain hardening during drawing

[0005] With the exception of structural strengthening, all the strengthening mechanisms cited above give rise to ductility loss in the final product.

[0006] The steels described in the above mentioned patents are based on a rather high content of hardenability increasing elements (Mo, Ni, Cr..) and thus are rather costly.

Summary of the Invention

[0007] It is an object of the invention to provide a low-carbon precipitation-strengthened bainitic steel in the form of hot rolled steel wire or rod that is comparatively cost-effective and at the same time is suitable for the production of cold headed products without the application of heat treating operations.

[0008] The foregoing and further objects are achieved by a hot rolled steel wire or rod having a chemical composition, by weight, of:

$$0.04\% \leq C \leq 0.1\%$$

$$1.8\% \leq Mn \leq 2.0\%$$

$$0.15\% \leq Si \leq 0.30\%$$

$$S \leq 0.025\%$$

$$P \leq 0.025\%$$

$$Cr \leq 0.50\%$$

$$Mo \leq 0.08\%$$

$$Ni+Cu \geq 0.30\%$$

$$0.01\% \leq Al \leq 0.05\%$$

$$V \leq 0.05\%$$

$$10\ ppm \leq B \leq 30\ ppm$$

$$N \leq 100\ ppm$$

$$Ti \geq 0.06\%$$

the remainder being iron and impurities resulting from the production process.

[0009] The steel has a cementite-free microstructure comprising a predominant phase, a minor phase and MX-precipitations distributed within the predominant phase, the predominant phase consisting of bainitic ferrite, the minor phase comprising retained austenite and optionally martensite with a size of 2 to 3 μm, the relative amount of the minor phase amounting to < 20% by volume.

[0010] According to metallurgical terminology for an MX particle, M represents metal atoms and X represents interstitial atoms, i.e., carbon and/or nitrogen. The MX particle could be a carbide, nitride or carbonitride particle. Generally, there are two types of MX particles: primary (large or coarse) MX particles and secondary (small or fine) MX particles. Primary MX particles in steel are usually greater than about 50 nm whereas secondary (small or fine) MX particles are usually less than about 20 nm. The conditions under which different metal atoms form MX particles vary with the composition of the steel alloy.

[0011] The absence of cementite, which is responsible for crack initiation in cold-deformed high-strength steels, makes the microstructure more resistant to void formation and failure. Some ductile films of austenite may also be intimately dispersed between ferrite which is advantageous due to their crack blunting effect.

[0012] According to another aspect of the invention, a method of producing the above defined steel wire or rod

comprises conventional normalized hot-rolling with a finish rolling temperature of ~1000°C and a subsequent cooling rate of > 3K/s between 800 and 500°C.

[0013] According to a further aspect of the invention, a method of producing the above defined steel wire or rod comprises a thermomechanically controlled process with a finish rolling temperature of ~800°C and a subsequent cooling rate of > 3K/s between 800 and 500°C.

[0014] According to yet another aspect of the invention, a steel wire or rod having the above defined chemical composition is used for producing a cold formable part.

[0015] Advantageous embodiments are defined in the dependent claims.

[0016] Surprisingly, it was found that a comparatively high titanium content of greater than 0.06 mass % in a low-carbon bainitic steel, preferably along with a proper thermomechanically controlled process (henceforth also abbreviated as "TMCP"), allows to develop a cementite-free granular bainite morphology with a limited volume fraction of less than 20% of a very fine second phase, which is most suitable for cold heading applications. Without being bound by theory, it appears that this type of morphology results from the austenite decomposition at higher transformation temperatures, below Bs temperature. Microalloying with high titanium content appears to have a special role in the formation of the granular bainite, since it separates the bainitic C-curve and provides also intense precipitation strengthening. The precipitation strengthening with very fine MX-carbonitrides appears to provide high strength of the rod or wire rod while decreasing the carbon content in the steel, which results in an increase in toughness and ductility.

[0017] By using a high titanium content of ≥ 0.06%, preferably in the range 0.1 to 0.2%, one can provide a contribution of approximately 200 MPa to the yield strength of the wire rod due to the precipitation of nano-particles of TiC in the bainitic ferrite. This allows reducing the carbon content in steels to below 0.10%, which enhances the workability in cold forming operations due to the limited amount of the second phase in the structure.

[0018] Another advantage of using a high titanium content is connected to the influence of this element on the morphology of bainite, namely, it promotes the development of granular bainite during accelerated cooling, preferably after TMCP. It was found that this effect is augmented by a proper application of low contents of alloying elements, such as Ni and Cu, which act synergistically to increase the carbon activity in bainitic ferrite and thus prevents the formation of cementite.

[0019] A basic level of the bainitic hardenability in the steel is achieved by using a high manganese content (~ 1.9%) and boron (0.0010 to 0.0030%).

[0020] A fine dispersion of the second phase in the wire rod is achieved by a proper selection of the finish rolling temperature. For product applications requiring high impact toughness at low temperatures, a finish rolling temperature in the range of 850 to 750°C should be applied. This produces extremely fine austenite grain size in the wire rod after the last pass, with mean linear intercept in the range of 10 to 15 μm being the prerequisite for the development of fine dispersion of the second phase with the size not exceeding 1 to 2 μm. For products not requiring high toughness at low temperature, static recrystallization control rolling can be applied with the finish rolling temperature of approximately 1000°C, which develops the fine austenite grain size with mean linear intercept in the range of 20 to 30 μm after the last pass. Again, the required austenite grain refinement appears to be achieved through the effect of titanium partly combined in the fine TiN particles and partly due to the dynamic precipitation of TiC during and after deformation.

[0021] The parameters of accelerated-controlled cooling conducted in the Stelmor line after the finish rolling is another prerequisite for achieving of the proper microstructure and mechanical properties in the wire rod. The fast cooling is performed with a rate in the range of 3 to 6°C/s (or even higher) and stopped at a temperature between 400 and 480°C.

[0022] The proper selection of the finish rolling temperature combined with an adjusted cooling practice and proper design of the bainitic steel chemical composition develops a very fine cementite-free granular bainite microstructure with bainitic ferrite strengthened by a dispersion of TiC nano-particles. Second phase particles in such a structure typically consists of a thin outer layer of M-A constituent and relatively ductile incomplete transformation reaction products inside. This provides a mechanical compatibility of the second phase during the deformation with the matrix, preventing the formation of microcracks. This morphology of the second phase is connected to the fact that the second phase in the granular bainite forms under the local equilibrium conditions with respect to carbon, prevailing at the bainitic ferrite/austenite interface, producing a steep carbon distribution profile in the residual austenite. Because an increase of the carbon content in the austenite lowers the martensite start temperature (Ms), the Ms temperature of the carbon enriched austenite may be lowered close to or below the room temperature.

[0023] The present invention provides the ways of achieving extremely high cold workability of the wire rod by using lean steel chemistries, and at the same time, allowing the achievement of high strength properties in the ultimate products. For example, for a wire rod with diameter of up to 20 mm, yield strength of approximately 550 to 800 MPa can be achieved, suitable for the production of fasteners with strength properties meeting 8.8 or even 10.9 grade requirements according to EN ISO 898 for fasteners due to the work hardening during cold deformation. It was found out that for bainitic steels drawing and cold heading operations could increase the strength properties by approximately 300 MPa.

[0024] The two main effects influencing the cold headibility in the invention are precipitation strengthening and developing granular bainite in the semi products. For that purpose, high titanium content is used in the bainitic steels being

the subject of the invention. Titanium promotes both the development of granular bainite morphology in a steel microstructure and a precipitation strengthening effect of up to 200 MPa, provided that the content of this element in a steel is greater than 0.06%, preferably in the range of 0.1 to 0.2%. To optimize the effect of this element on the morphology of granular bainite and precipitation strengthening, TMCP combined with a proper cooling conditions after rolling must be used.

[0025] It is noted that none of the prior art disclosures addressed directly the most suitable type of bainitic microstructure and the role of carbide forming elements, such as Ti, V and Nb, as well as the thermomechanical control process (TMCP) in shaping of this type of microstructure, workability during the cold forging and mechanical properties of ultimate products. The role of the microalloying additions used in the steels disclosed in the prior art publications is not precisely defined, which suggests that their use is mainly intended to control the austenite microstructure evolution during the rolling process.

Brief description of the drawings

[0026] The above mentioned and other features and objects of this invention and the manner of achieving them will become more apparent and this invention itself will be better understood by reference to the following description of various embodiments of this invention taken in conjunction with the accompanying drawings, wherein:

Fig. 1 shows a micrograph of a first steel heat ("Heat 1180") after cooling to 400°C without cold deformation;

Fig. 2 shows a micrograph of a second steel heat ("Heat 1181") after cooling to 400°C without cold deformation; and

Fig. 3 shows a micrograph of a third steel heat ("Heat 1209") after final rolling at 850°C.

Detailed description of the invention

Example 1: conventional normalized hot rolling

[0027] Two steel heats (denoted here as "1180" and "1181") were subjected to conventional normalized hot rolling. The chemical compositions of the two steels are shown in Table 1:

Table 1: Chemical compositions in mass percentage

Heat	C [%]	Mn [%]	Si [%]	P [%]	s [%]	Ni [%]	Cu [%]	Ti [%]	Al [%]	B [%]
1180	0.052	1.87	0.21	0.014	0.014	0.15	0.16	0.10	0.026	0.002
1181	0.08	1.95	0.20	0.012	0.015	0.20	0.21	0.10	0.028	0.003

[0028] The initial sample geometry was 45 x 45 x 150 mm. The ingots were heated up in a furnace for 15 min at 1200°C. Then the bars were rough rolled from the initial geometry to a wire of 12 mm in diameter. The rolling was performed in 12 steps. After the rough rolling, the finish rolling was performed continuously in four steps down to a diameter of 8 mm. The material temperature before the first continuous pass was 870°C, and before the last continuous pass it was 980°C. The increase in the temperature is due to the deformation heat.

[0029] The finishing rolling speed was 20 m/s. The cooling was conducted on a Stelmor line at full ventilation speed. The cooling rate between 800°C and 500°C was 8 K/s. The cooling stop temperature was set to 400°C. The mechanical properties were determined in the hot rolled as well as in the drawn condition (see Table 2). The drawing reduction was 10% in one pass.

Table 2: Mechanical properties

Steel heat / cooling stop temperature / processing	Sample No	Yield strength $R_{p0.2}$ [MPa]	Tensile strength R_m [MPa]	Elongation at fracture A_{25} [%]	Reduction of area Z [%]
1180/400°C/as-hot rolled	1	722	915	27.9	75.1
	2	766	930	28.1	75.9
	3	802	911	27.6	74.7

(continued)

Steel heat / cooling stop temperature / processing	Sample No	Yield strength $R_{p0.2}$ [MPa]	Tensile strength R_m [MPa]	Elongation at fracture A_{25} [%]	Reduction of area Z [%]
1180/400°C/ cold drawn	1	950	991	26.6	74.0
	2	941	993	26.4	73.9
	3	953	996	26.4	-
1181/400°C/as-hot rolled	1	630	800	27.9	66.6
	2	641	805	27.7	71.1
	3	680	744	-	71.2
1181/400°C/ cold drawn	1	863	903	27.3	72.3
	2	810	884	27.1	67.5
	3	788	851	27.8	68.6

[0030] Figures 1 and 2 show the typical structure after hot rolling.

Example 2: *Thermomechanically controlled process (TMCP)*

[0031] A further steel heat (denoted here as "1209") was subjected to a thermomechanically controlled hot rolling process. The chemical composition of the steel is shown in Table 3:

Table 3: Chemical compositions in mass percentage

Heat	C[%]	Mn [%]	Si [%]	P [%]	S [%]	Ni [%]	Cu [%]	Ti [%]	Al [%]	B [%]
1209	0.08	1.78	0.19	0.011	0.009	0.16	0.16	0.10	0.029	0.002

[0032] The initial sample geometry was 45 x 45 x 150 mm. The ingots were heated up in the furnace for 15 min at 1200°C. Then the bars were rough rolled from the initial geometry to a wire of 12 mm in diameter. The rolling was performed in 12 steps. Unlike the conventional rolling experiments of example 1, the rolling was stopped after the reversing mill and the rods were directly cooled in the cooling device with air. At the start of rolling the temperature was 1180°C, and the finishing temperature would have been around 1000°C. In order to carry out a rolling below the recrystallisation temperature, the rolling was interrupted for several seconds until the rod temperature was 850°C. Subsequently, the last pass was conducted. The cooling rate between 800°C and 500°C was 5.5 K/s.

[0033] The rolling below RST (recrystallisation stop temperature) led to a pancaked austenite microstructure which was inherited in the final structure as well (Figure 3).

[0034] The mechanical properties were determined in the hot rolled condition (see Table 4).

Table 4: Mechanical properties (heat 1209)

State	"$R_{p0.2}$ [MPa]	R_m [MPa]	A_g [%]	A_t [%]	Z [%]
Air cooled 1	559	784	9.1	19.2	72
Air cooled 2	543	782	9.1	18.7	75
Air cooled 3	529	782	9.4	19.1	69

[0035] The Charpy impact toughness measured during the tests conducted on non-standard samples was very high (see Table 5). This was not the case when finish rolling temperature was above RST.

Table 5: Impact toughness of wire rod (heat 1209)

Sample No	Impact toughness at -20° [J/cm^2]	Impact toughness at -40°C [J/cm^2]
L1	145	-
L2	135	-
L3	140	-
L4		135
L5		144
L6		132

[0036] In the hot-rolled condition all steels (conventionally normalized and thermomechanically hot rolled) show a high tensile strength combined with high ductility and excellent reduction of area values which is advantageous for cold heading.

[0037] Based on the mechanical properties in the as-rolled condition 8.8 or even 10.9 grade requirements according to EN ISO 898 can be achieved by work hardening during drawing and cold heading without any final heat treatment.

[0038] Only cold drawing of the steel causes an increase of the yield strength of approx. 200 MPa and of the tensile strength of approx. 50 to 100 MPa while there is no substantial loss with regard to the elongation at fracture and reduction of area.

[0039] The structure of the steel subjected to a thermomechanically controlled hot rolling process (heat 1209) has a microstructure that is more resistant to crack propagation, thus enabling very high high impact toughness to be achieved at low temperatures.

Claims

1. A rolled steel wire or rod, **characterized in** having a chemical composition, by weight, of:

$$0.04\% \leq C \leq 0.1\%$$

$$1.8\% \leq Mn \leq 2.0\%$$

$$0.15\% \leq Si \leq 0.30\%$$

$$S \leq 0.025\%$$

$$P \leq 0.025\%$$

$$Cr \leq 0.50\%$$

$$Mo \leq 0.08\%$$

$$Ni+Cu \geq 0.30\%$$

$$0.01\% \leq Al \leq 0.05\%$$

$$V \leq 0.05\%$$

$$10\ ppm \leq B \leq 30\ ppm$$

$$N \leq 100\ ppm$$

$$Ti \geq 0.06\%$$

the remainder being iron and impurities resulting from the production process,
with a cementite-free microstructure comprising a predominant phase, a minor phase and MX-precipitations distributed within the predominant phase, the predominant phase consisting of bainitic ferrite, the minor phase comprising retained austenite and optionally martensite with a size of 2 to 3 μm, the relative amount of the minor phase amounting to < 20% by volume.

2. The rolled steel wire or rod according to claim 1, **characterized in** having the following mechanical properties at 20°C:

a yield strength (Rp0.2) of 550 to 800 MPa,
a tensile strength (Rm) of 700 to 1000 MPa,
an elongation at fracture (A25) of 15 to 30%, and
a reduction of area (Z) of 60 to 80%.

3. A method of producing the steel wire or rod according to claim 1 by conventional normalized hot-rolling with a finish rolling temperature of ~1000°C and a subsequent cooling rate of > 3K/s between 800 and 500°C.

4. A method of producing the steel wire or rod according to claim 1 by a thermomechanically controlled process with a finish rolling temperature of ~800°C and a subsequent cooling rate of > 3K/s between 800 and 500°C.

5. Use of a steel with a chemical composition, by weight, of:

$$0.04\% \leq C \leq 0.1\%$$

$$1.8\% \leq Mn \leq 2.0\%$$

$$0.15\% \leq Si \leq 0.30\%$$

$S \leq 0.025\%$

$P \leq 0.025\%$

$Cr \leq 0.50\%$

$Mo \leq 0.08\%$

$Ni+Cu \geq 0.30\%$

$0.01\% \leq Al \leq 0.05\%$

$V \leq 0.05\%$

$10\ ppm \leq B \leq 30\ ppm$

$N \leq 100\ ppm$

$Ti \geq 0.06\%$

the remainder being iron and impurities resulting from the production process, for producing a part by cold forming.



Fig. 1

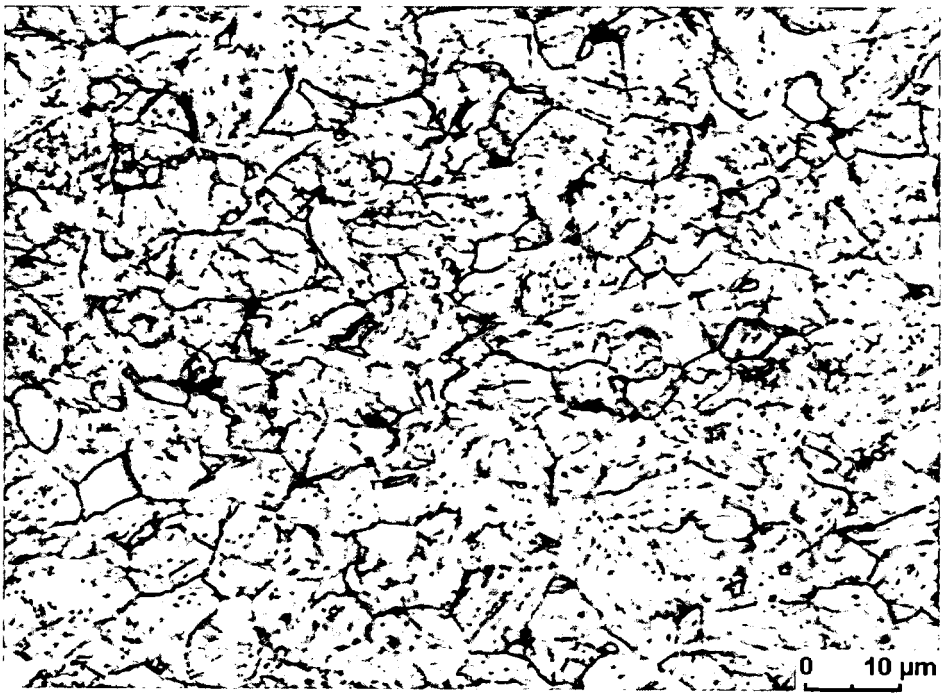


Fig. 2

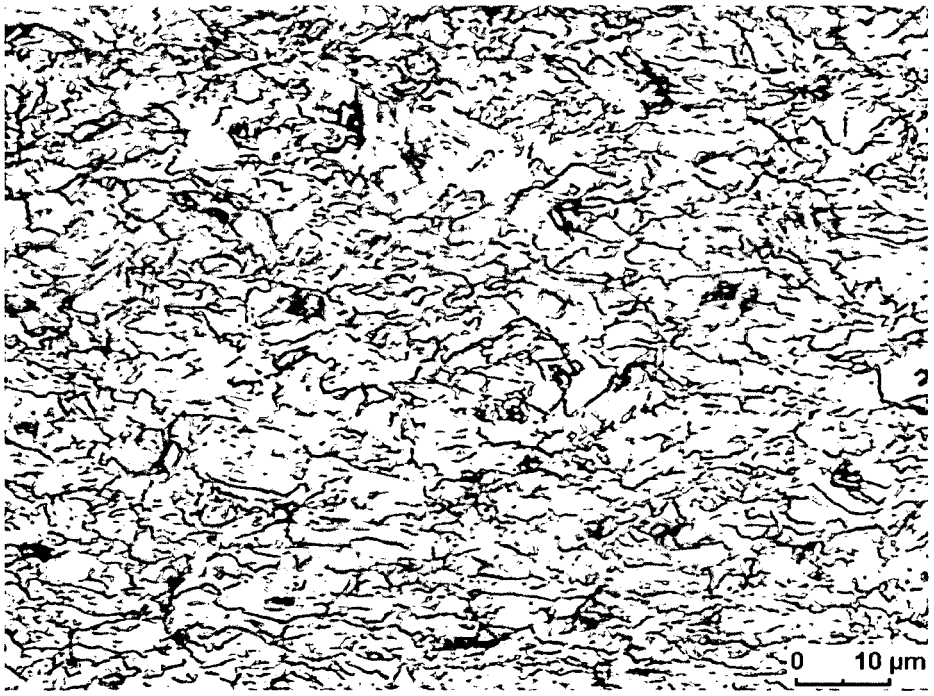


Fig. 3

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EUROPEAN SEARCH REPORT

Application Number
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The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
The Hague	9 June 2009	Vlassi, Eleni

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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A : technological background
O : non-written disclosure
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EUROPEAN SEARCH REPORT

Application Number
EP 08 02 1703

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D : document cited in the application
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ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 08 02 1703

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