



(86) Date de dépôt PCT/PCT Filing Date: 2011/03/23
 (87) Date publication PCT/PCT Publication Date: 2011/12/08
 (45) Date de délivrance/Issue Date: 2016/05/10
 (85) Entrée phase nationale/National Entry: 2012/11/30
 (86) N° demande PCT/PCT Application No.: FR 2011/000167
 (87) N° publication PCT/PCT Publication No.: 2011/151532
 (30) Priorité/Priority: 2010/05/31 (FR1002286)

(51) Cl.Int./Int.Cl. *C21D 9/52* (2006.01),
C21D 1/20 (2006.01), *C21D 9/58* (2006.01),
C22C 38/02 (2006.01), *C22C 38/04* (2006.01)

(72) Inventeurs/Inventors:
 FOISSEY, SYLVAIN, FR;
 BERTOUT, CHRISTOPHE, FR;
 PERROUD, XAVIER, FR

(73) Propriétaire/Owner:
 ARCELORMITTAL WIRE FRANCE, FR

(74) Agent: SMART & BIGGAR

(54) Titre : FIL DE FORME EN ACIER A HAUTES CARACTERISTIQUES MECANIQUES RESISTANT A LA
 FRAGILISATION PAR L'HYDROGENE

(54) Title: PROFILED STEEL WIRE WITH HIGH MECHANICAL CHARACTERISTICS RESISTANT TO HYDROGEN
 EMBRITTLEMENT

(57) **Abrégé/Abstract:**

This profiled wire, of NACE grade, made of low-alloy carbon steel intended to be used in the offshore oil exploitation sector, is characterized in that it has the following chemical composition, expressed in percentages by weight of the total mass: $0.75 < \% C < 0.95$; $0.30 < \% Mn < 0.85$; $Cr \leq 0.4\%$; $V \leq 0.16\%$; $Si \leq 1.40\%$ and preferably $\geq 0.15\%$; and optionally no more than 0.06% Al, no more than 0.1% Ni and no more than 0.1% Cu, the balance being iron and the inevitable impurities arising from smelting the metal in the liquid state, and in that the steel is obtained, from hot-rolled rod stock cooled down to room temperature, and then having a diameter of about 5 to 30 mm, by subjecting this starting rod firstly to a thermomechanical treatment comprising two successive steps carried out in order, namely an isothermal quench, giving it a homogeneous perlitic microstructure, followed by a mechanical transformation operation carried out cold with an overall degree of work-hardening (or reduction ratio) of between 50 and 80% at most, so as to give the wire its definitive shape, and in that the profiled wire thus obtained is then subjected to a restoration heat treatment of short duration carried out below Ac1 (preferably between 410 and 710°C), giving it the desired final mechanical properties.

(12) DEMANDE INTERNATIONALE PUBLIÉE EN VERTU DU TRAITÉ DE COOPÉRATION EN MATIÈRE DE BREVETS (PCT)

(19) Organisation Mondiale de la Propriété Intellectuelle
Bureau international(43) Date de la publication internationale
8 décembre 2011 (08.12.2011)

PCT

(10) Numéro de publication internationale
WO 2011/151532 A1

- (51) Classification internationale des brevets :
C21D 9/52 (2006.01) C21D 1/20 (2006.01)
C22C 38/04 (2006.01) C21D 9/58 (2006.01)
C22C 38/02 (2006.01)
- (21) Numéro de la demande internationale :
PCT/FR2011/000167
- (22) Date de dépôt international :
23 mars 2011 (23.03.2011)
- (25) Langue de dépôt : français
- (26) Langue de publication : français
- (30) Données relatives à la priorité :
1002286 31 mai 2010 (31.05.2010) FR
- (71) Déposant (pour tous les États désignés sauf US) :
ARCELORMITTAL WIRE FRANCE [FR/FR]; 25,
Avenue de Lyon, BP 96, F-01000 Bourg en Bresse (FR).
- (72) Inventeurs; et
- (75) Inventeurs/Déposants (pour US seulement) : FOISSEY,
Sylvain [FR/FR]; 6, rue de Bressy, F-01800 Meximieux
(FR). BERTOOUT, Christophe [FR/FR]; 249, Avenue de
Macon, F-01440 Viriat (FR). PERROUD, Xavier
[FR/FR]; Chemin de la Côtière, F-01800 Mollon (FR).
- (74) Mandataire : ARCELORMITTAL France; Arcelor
Research Industrial Property (ARIP), 5 rue Luigi
Cherubini, 93312 La Plaine Saint Denis Cedex (FR).
- (81) États désignés (sauf indication contraire, pour tout titre
de protection nationale disponible) : AE, AG, AL, AM,
AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ,
CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO,
DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT,
HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP,
KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD,
ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI,
NO, NZ, OM, PE, PG, PH, PL, PT, RO, RS, RU, SC, SD,
SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR,
TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.
- (84) États désignés (sauf indication contraire, pour tout titre
de protection régionale disponible) : ARIPO (BW, GH,
GM, KE, LR, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG,
ZM, ZW), eurasien (AM, AZ, BY, KG, KZ, MD, RU, TJ,
TM), européen (AL, AT, BE, BG, CH, CY, CZ, DE, DK,
EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU,
LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK,
SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ,
GW, ML, MR, NE, SN, TD, TG).
- Déclarations en vertu de la règle 4.17 :**
- relative au droit du déposant de demander et d'obtenir un brevet (règle 4.17.ii)
 - relative à la qualité d'inventeur (règle 4.17.iv)
- Publiée :**
- avec rapport de recherche internationale (Art. 21(3))

(54) Title : PROFILED WIRE MADE OF HYDROGEN-EMBRITTEMENT-RESISTANT STEEL HAVING HIGH MECHANICAL PROPERTIES

(54) Titre : FIL DE FORME EN ACIER À HAUTES CARACTÉRISTIQUES MÉCANIQUES RÉSISTANT À LA FRAGILISATION PAR L'HYDROGÈNE

(57) Abstract : This profiled wire, of NACE grade, made of low-alloy carbon steel intended to be used in the offshore oil exploitation sector, is characterized in that it has the following chemical composition, expressed in percentages by weight of the total mass: 0.75 < % C < 0.95; 0.30 < % Mn < 0.85; Cr ≤ 0.4%; V ≤ 0.16%; Si ≤ 1.40% and preferably ≥ 0.15%; and optionally no more than 0.06% Al, no more than 0.1% Ni and no more than 0.1% Cu, the balance being iron and the inevitable impurities arising from smelting the metal in the liquid state, and in that the steel is obtained, from hot-rolled rod stock cooled down to room temperature, and then having a diameter of about 5 to 30 mm, by subjecting this starting rod firstly to a thermomechanical treatment comprising two successive steps carried out in order, namely an isothermal quench, giving it a homogeneous perlite microstructure, followed by a mechanical transformation operation carried out cold with an overall degree of work-hardening (or reduction ratio) of between 50 and 80% at most, so as to give the wire its definitive shape, and in that the profiled wire thus obtained is then subjected to a restoration heat treatment of short duration carried out below Ac1 (preferably between 410 and 710°C), giving it the desired final mechanical properties.

(57) Abrégé : Ce fil de forme, de qualité NACE, en acier au carbone faiblement allié destiné à être utilisé dans le secteur de l'exploitation pétrolière off shore, se caractérise en ce qu'il présente la composition chimique suivante, exprimée en pourcentages pondéraux de la masse totale, le reste étant du fer et les impuretés inévitables venant de l'élaboration du métal à l'état liquide: 0,75 < C % < 0,95 et 0,30 < Mn % < 0,85 avec Cr ≤ 0,4%; V ≤ 0,16%; Si ≤ 1,40% et de préférence ≥ 0,15%; et éventuellement pas plus de 0,06% d'Al, pas plus de 0,1% de Ni, et pas plus de 0,1% de Cu, et en ce qu'il est obtenu, à partir d'un fil machine, laminé à chaud et refroidi à la température ambiante, et présentant alors un diamètre de 5 à 30 mm environ, en soumettant ce fil de départ d'abord à un traitement thermomécanique selon deux étapes successives et ordonnées, à savoir une trempe isotherme qui lui confère une microstructure perlitique homogène, suivie par une opération de transformation mécanique à froid menée avec un taux d'érouissage global compris entre 50 et 80% maximum pour donner au fil sa forme définitive, et en ce que le fil de forme ainsi obtenu est alors soumis à un traitement thermique de restauration de courte durée mené en dessous d'Ac1 (de préférence entre 410 et 710 °C), lui conférant les caractéristiques mécaniques finales désirées.

WO 2011/151532 A1

**Profiled steel wire with high mechanical characteristics
resistant to hydrogen embrittlement**

The present invention concerns the field of metallurgy dedicated to maritime oil well exploitation. More particularly, it deals with steel wires that can be used as strengthening or structural elements of components or works submerged in deep water, such as flexible offshore pipelines.

We know that a first requirement with regard to this type of wires, besides elevated mechanical characteristics (in particular Ultimate Tensile Strength), is good resistance to hydrogen embrittlement in a sulfuric acid medium and especially in the form of the H₂S present in the fluids and hydrocarbons that are transported.

One is reminded that this resistance is the subject of NACE and API standards, in particular:

- NACE standard TM 0284 for the Hydrogen Induced Cracking (HIC) in sea water saturated with acidic H₂S;
- NACE standard TM 0177 for Sulfide Stress Corrosion Cracking (SSCC) in an acidic environment. Profiled wires, in the use considered here, must absolutely deal today with increasingly more difficult operating conditions (great depth);
- and API standard 17J (Specification for unbonded flexible pipes) for evaluation of the HIC and SSCC behavior on the basis of a stress test in an acidic environment.

These profiled wires can have a round cross section, obtained by plain drawing from a wire rod of greater diameter. They can also have, after drawing, rolling, or drawing followed by rolling, have a rectangular section, or be profiled in a U, a Z, a T, etc., so as to be able to fit together by their edges or be stapled together to form linked reinforcement mats.

At present, the commercial offering in the field of NACE grade steel wires for offshore use lies primarily in low-alloy steel grades which ultimately provide, after quenching and tempering, a ultimate tensile strength (R_m) of around 900 MPa.

To fabricate these profiled wires one generally uses, as is known, carbon manganese steels of 0.15-0.80% C (by weight), having an initial perlite-ferrite structure. Classically, after shaping the initial round rolled wire rod, one applies a heat treatment of suitable duration to obtain the desired strength. It is this hardness level for which the nominal criteria of use are observed, for example, standard ISO 15156, stipulating that these grades of Mn steel have a stress resistance in H₂S environment suitable for the "profiled wire" use in question if the hardness of the wire is less than or equal to 22 HRC.

However, the profiled wires obtained by the traditional methods have the reputation of being ill suited to withstand relatively harsh conditions of acidity such as the one encountered in deep waters, in the present instance, those set forth by the NACE standard TM 0177 with solution A (pH of 2.7 to 4), due to a heavy presence of H₂S in the hydrocarbon being transported, and all the more so if said hardness levels are greater than 28 HRC (more than 900 MPa).

Furthermore, this is doubtlessly the reason why document PCT/FR91/00328, published in 1991, describes a thermomechanical method for production of a profiled wire of perlite-ferrite structure having between 0.25 and 0.8% carbon and meeting the NACE TM 0177 and TM 0284 standards with solution B (pH 4.8 to 5.4), yet at the cost of a final annealing to relieve the mechanical stresses imprinted by the work hardening of the metal, which lowers the ultimate tensile strength (R_m) to around 850 MPa.

Document FR-B-2731371, published in 1996, also deals with the production of profiled wires of carbon steel for the reinforcement of offshore flexible pipelines whose strength in acidic environment with H₂S is sought at an elevated level thanks to general knowledge as to the influence of steel microstructures on its resistance to hydrogen embrittlement. The profiled wire proposed in this document, which contains from 0.05 to 0.8% of C and from 0.4 to 1.5% of Mn, has undergone, after shaping (drawing or drawing-rolling), a quenching followed by a final tempering. The metallic structure obtained is essentially annealed martensite-bainite. One thus would obtain ready-to-use profiled wires having elevated mechanical characteristics, namely, a R_m near 1050 MPa (thus, in a quenched and annealed steel, to obtain hardness levels as high as 35 HRC, but industrially determined in fact around 820 MPa) and consequently clearly beyond those recommended by the standard ISO 15156, and resistant to very acidic environments (pH near 3). It is noted that, without a final annealing, one can obtain a wire with a greater hardness, having even more elevated mechanical characteristics, but then with a distinctly lower chemical resistance to acid environments.

In fact, one finds that the characteristics of very elevated level afforded by such wires only need to be met in a limited number of application instances. According to the NACE grade, a strength according to the aforesaid API 17J standard, with a partial H₂S pressure reaching 0.1 bar and with a pH of 3.5 to 5, would in fact be sufficient to handle the basic requirements, whereas the profiled wires fabricated by the method of the cited document have so to speak an overqualified strength, since they conform to the elevated demands of the TM 0177 and TM 0284 standards, which are established with solution A having a pH of around 3.

Furthermore, it turns out that the customary profiled wires on the market, of perlite-ferrite structure with no final heat treatment, are for the most part ill suited to meet even modest NACE demands.

What is more, the flexible offshore pipelines being called upon to serve ever greater depths of immersion, there is a distinct demand for an even greater strength by a couple of hundred MPa, to reach strengths on the order of, say, 1300 MPa, or even more, without thereby degrading the NACE quality, whereas we should keep in mind that hydrogen embrittlement of steel and mechanical characteristics are opposite properties: to boost one of them comes at the expense of the other, and vice versa.

Furthermore, market constraints are constantly rising in terms of price, which accordingly hampers the customary solution of using noble alloy elements, such as chromium, niobium, etc, or of using long or multiple treatment operations, which are costly in particular if made at high temperatures.

To this extent, one must take into account the teaching of DATA BASE WPI Week 198407 Thomson Scientific, London, GB, AN 1984-039733 proposing a final and long recovery heat treatment of the wire consisting in an annealing lasting several hours.

Moreover, the method described in EP 1 063 313 A1 imposes very high work hardening rates, around 85%, to obtain a drawing of the wire to the targeting final diameter.

One must also take into account EP 1 273 670 dealing with the manufacturing of steel bolts, but which teaching underlines the interest that can be awaited for the tension corrosion resistance of politic bolts.

The invention proposes to achieve an optimal equilibrium between a required good resistance to wet hydrogen embrittlement under conditions of use of the profiled wire and its enhanced mechanical strength, in the frame of an industrial production allowing proposing the wire to the market, at attractive economic conditions.

According to various aspects, the present invention relates to a process for making a profiled wire of hydrogen-embrittlement-resistant, low-alloy carbon steel, the process comprising the step of: hot-rolling a wire rod in its austenitic domain above 900°C to obtain a diameter between 5 to 30 mm; cooling down the wire rod to room temperature; subjecting the wire rod to a thermo-mechanical treatment by two consecutive and ordered phases: an isothermal tempering to confer on the wire rod a homogeneous perlite microstructure, followed by; a cold mechanical transformation operation with an overall work hardening rate comprised between 50% and 80% max, to give the wire rod a final shape; and subjecting the profiled wire rod to a heat treatment at a temperature from 410°C to 719°C for a duration of one minute or less.

According to various aspects, the present invention relates to a process for making a profiled wire of hydrogen-embrittlement-resistant, low-alloy carbon steel, the process comprising the step of: hot-rolling a wire rod in its austenitic domain above 900°C to obtain a diameter between 5 to 30 mm; cooling down the wire rod to room temperature; subjecting the wire rod to a thermo-mechanical treatment by two consecutive and ordered

3a

phases: an isothermal tempering to confer on the wire rod a homogeneous perlite microstructure, followed by; a cold mechanical transformation operation with an overall work hardening rate comprised between 50% and 80% max, to give the wire rod a final shape; and subjecting the profiled wire rod to a heat treatment at a temperature from 410°C to 719°C for a duration of one minute or less; wherein, the profiled wire is suitable for use as a flexible tube component in offshore oil well drilling and comprising in percent by weight of the total mass

$$0.75 \leq C \% \leq 0.95 \text{ and}$$

$$0.30 \leq Mn \% \leq 0.8$$

with $Cr \leq 0.4\%$; $V \leq 0.16\%$; $Si \leq 1.40\%$, and possibly not more than 0.06% Al, not more than 0.1% Ni and not more than 0.1% Cu, the remainder being iron and impurities resulting from processing of metal in a liquid state.

For this, the invention concerns a profiled wire made of low-alloy carbon steel with high mechanical properties and resistant to hydrogen embrittlement, profiled wire intended for use as flexible tube component in the offshore oil well drilling sector, characterized in that it has the following chemical composition, given in percent by weight of the total mass, the remainder being iron and the unavoidable impurities resulting from processing of metal in the liquid state:

$$0.75 \leq C \% \leq 0.95 \text{ and}$$

$$0.30 \leq Mn \% \leq 0.85$$

with $Cr \leq 0.4\%$; $V \leq 0.16\%$; $Si \leq 1.40\%$ and preferably $\geq 0.15\%$;

and optionally not more than 0.06% of Al, not more than 0.1% of Ni, and not more than 0.1% of Cu, and in that, starting from a wire rod, hot-rolled in its austenitic domain above 900°C and cooled down to room temperature to have a 5 to 30 mm diameter, the profiled wire is obtained by first subjecting said starting wire rod to a thermomechanical treatment by two consecutive and ordered phases, namely, an isothermal tempering (classically, a lead patenting) to confer on the wire rod a homogeneous perlite microstructure, followed by a cold mechanical transformation operation (drawing, or drawing + rolling) with an overall work hardening rate comprised between 50 and 80% max (and, if possible, preferably around 60%), to give it its final shape, and in that the obtained profiled wire is then subjected to a recovery heat treatment of short duration (preferably less than one minute), at a temperature lower than the Ac1 temperature of the steel of which it is made (preferably from 410 to 710°C), giving it the desired final mechanical characteristics.

According to various aspects, the present disclosure relates to a process for making a profiled wire of hydrogen-embrittlement-resistant, low-alloy carbon steel, the process comprising the following consecutive steps:

- hot-rolling a wire rod in its austenitic domain above 900°C to obtain a diameter between 5 to 30 mm;
- cooling down the wire rod to room temperature;
- subjecting the wire rod to a thermo-mechanical treatment by two consecutive and ordered phases:
 - an isothermal tempering to confer on the wire rod a homogeneous perlite microstructure, followed by;
 - a cold mechanical transformation operation with an overall work hardening rate comprised between 50% and 80% max, to give the wire rod a final shape; and
- subjecting the profiled wire rod to a heat treatment at a temperature from 410°C to 719°C for a duration of one minute or less;

wherein, the profiled wire is suitable for use as a flexible tube component in offshore oil well drilling and comprising in percent by weight of the total mass,

$$0.75 \leq C \% \leq 0.95;$$

$$0.30 \leq Mn \% \leq 0.85;$$

$$Cr \leq 0.4\%;$$

$$V \leq 0.16\%;$$

$$Si \leq 1.40\%; \text{ and}$$

the remainder being iron and impurities resulting from processing of metal in a liquid state.

4a

The invention which has just been defined above is based on three elements: steel grade, treatment, application and can be viewed as an optimization of the knowledge gained by the applicant in the field of the metallurgy of steel wires intended to be used in the deep sea.

More explicitly, these three elements can be detailed as follows:

- a simplified steel grade, that is, a carbon (at least 0.75%) and manganese steel, which is thus contrary to the much lower carbon contents currently used, and without adding hardening elements, but preferably alloyed with dispersoid elements, such as vanadium and chromium, to obtain a homogeneous distribution of fine carbides in the entire metal matrix;

- this grade is produced from a hot-rolled wire rod subsequently cooled down to room temperature (i.e., having an ordinary ferrite-perlite structure Inherited from the austenite present at the hot-rolling stage), but whose diameter (between 5 and 30 mm) is reduced as compared to the usual practice. This feature will enable Its final transformation into a ready-to-use profiled wire by soft mechanical shaping operations, that is, without a significant work hardening throughout, which might create zones of heterogeneity, noting that the operator in charge of the manufacturing process will have to adjust the operating parameters (adjusting of operational parameters, choice of draw plates and grooves of the rolls) to limit the local work hardening inside the wire.

The microstructure to be created by the isothermal tempering is perlite. Perlite, which is easy to produce industrially, will ensure the most homogeneous possible metallurgical structure in the entire mass of the wire produced and it will be able to withstand the deformations applied by drawing and/or rolling.

- this wire is a wire with a flat shape or a shape including flat parts, or profiled, intended for offshore oil well drilling use to form the winding, hoop or arch wire in the structure of flexible pipelines or other pipes. As is known, profiled steel wires advance in the pipelines between two layers of extruded polymer, in a so-called "annular" zone. The physicochemical conditions prevailing in this zone during the use of the flexible pipeline are better known at present. They depend on the nature of the effluent in the pipeline (liquid or gaseous hydrocarbons) and the structure of the different layers of the pipeline. In particular, the pH is higher than was believed in 1990/2000 (on average more like 5.5 than 4). Thus, the invention finds its purpose in the discovery of these new, less drastic conditions to be satisfied in the annular zone, which allows for the use of profiled wires with higher mechanical strength.

In other words, the NACE quality of today can be expressed quite validly through less demanding test results than those of the API standard (the applicant was thus forced to adapt the test conditions as compared to the API standard, especially the pH, in order to adapt to the demand). For example, the NACE quality can be assigned to a steel wire having withstood without breaking or internal cracking for one month under a continual stress of 90% of Re in an aqueous solution having a pH between 5 and 6.5 and subjected to bubbling of a gas containing CO₂ and several millibars of H₂S.

The invention will be better understood and other aspects and advantages will appear more clearly in light of the following description, given as an example.

Table I, shown on the last page of this specification, shows seven examples of chemical compositions of grades according to the invention, as is found in the first column using the internal nomenclature of the applicant.

We shall now discuss in detail one exemplary composition example in the steel grade referenced as C88 (next to last row of table I), whose components correspond to the following weight contents: C: 0.861%, Mn: 0.644%, P: 0.012%, S: 0.003%, Si: 0.303%, Al: 0.47%, Ni: 0.015%, Cr: 0.032%, Cu: 0.006%, Mo: 0.003%, and V: 0.065%.

Starting from a round wire rod of 12 mm diameter having this composition, one makes a final ready-to-use wire with a shape including flat parts, 9 mm x 4 mm, by the following consecutive operations.

Let it first be noted that, according to the invention, a diameter of 30 mm will not be exceeded for the initial wire rod, so as not to have to work the core of the wire to a substantial degree during the subsequent drawing made with a global work hardening rate of 80% max, in order to reach the final diameter of the ready-to-use profiled wire.

The wire rod is a steel wire hot-rolled in its austenitic domain (typically above 900°C) that has been rapidly cooled down in the rolling heat, before being wound into a coil to end up its cooling down to room temperature in a storage area, waiting to be delivered to the customer.

Once delivered to the customer, this starting wire rod that is unwound from its reel first undergoes, from the room temperature, an isothermal tempering. Typically, it will consist in a patenting at constant temperature of around 520-600° C by going through a molten lead bath, prior to cooldown. The patenting operation confers to the steel wire a perlite microstructure, with possible traces of ferrite, but with no bainite or martensite, and which it will preserve till the end.

The wire is then drawn (round or already partially flattened) in a "soft" way, that is, as already mentioned above, so as to limit to the maximum the level of internal stresses produced by the working of the metal. The reason for this is to limit the damage to the internal microstructure, which damage would create sites favorable to a preferential accumulation of hydrogen. The wire can then undergo a cold rolling to reach the final dimensions, its being noted that the overall work hardening (drawing + rolling) rate will be from 50 to 80% max, and, if possible preferably around 60%.

The intermediate wire thus obtained has a R_m of around 1900 MPa.

It remains to soften it to facilitate its later shaping, and give it its properties of resistance to hydrogen embrittlement, impaired by the work hardening. For this purpose, a simple final recovery heat treatment, i.e., at a temperature below its A_{c1} value (from 410 to 710°C for the steel grades used) and lasting less than one minute, will give it the final R_m desired, whose exact value will depend, of course, on the operating conditions of this recovery treatment.

In this context, table II below gives the final mechanical characteristics obtained for a profiled wire having undergone a recovery heat treatment under the following operating conditions, designated by lines A to E: holding for a time of 5 seconds at a temperature less than the A_{c1} temperature of the grade considered and given in the second column of the table, before sudden cooling with water.

The other columns show respectively the mean ultimate tensile strength R_m , the mean elastic limit R_e , the mean rate of elongation at breakage $A\%$ of the treated wire resulting from the thermomechanical operations carried out, and the ratio R_e/R_m .

It will be noted, as might be expected, that R_m , like R_e , decreases regularly as the recovery temperature rises (rows A to E). The ratio R_e/R_m remains constant and the rate of elongation $A\%$ increases in the same direction.

Table II

	Recovery temp. (°C)	Mean Rm (MPa)	Mean Re (MPa)	Mean A%	Re/Rm
A	410	1920	1730	9.6	0.90
B	500	1760	1530	9.7	0.86
C	600	1550	1360	11.0	0.87
D	635	1480	1280	12.0	0.86
E	675	1380	1190	11.6	0.86

The NACE tests, by the HIC (Hydrogen Induced Cracking) and SSC (Sulfide Stress Cracking) mode, were performed on each of the wires obtained after these different recovery heat treatments. The data and the results are shown in table III below.

One sees that all the samples analyzed pass the tests: after ultrasound inspection, one finds no internal cracks of blister type, which would indicate an embrittlement by hydrogen corrosion.

Table III

	Rm (in MPa)	NACE test type	Duration (in days)	H ₂ S, %	pH	Stress applied in SSC	US Results
A	1920	HIC + SSC	30	0.1	5.8	90% Re	RAS
B	1760	HIC + SSC	30	0.1	5.8	90% Re	RAS
C	1550	HIC + SSC	30	0.22	5.6	90% Re	RAS
D	1480	HIC + SSC	30	0.22	5.6	90% Re	RAS
E	1380	HIC + SSC	30	0.22	5.6	90% Re	RAS

The scope of the claims should not be limited by the preferred embodiments set forth in the examples, but should be given the broadest interpretation consistent with the description as a whole.

CLAIMS:

1. A process for making a profiled wire of hydrogen-embrittlement-resistant, low-alloy carbon steel, the process comprising the following consecutive steps:

- hot-rolling a wire rod in its austenitic domain above 900°C to obtain a diameter between 5 to 30 mm;
- cooling down the wire rod to room temperature;
- subjecting the wire rod to a thermo-mechanical treatment by two consecutive and ordered phases:
 - o an isothermal tempering to confer on the wire rod a homogeneous perlite microstructure, followed by;
 - o a cold mechanical transformation operation with an overall work hardening rate comprised between 50% and 80% max, to give the wire rod a final shape; and
- subjecting the profiled wire rod to a heat treatment at a temperature from 410°C to 719°C for a duration of one minute or less;

wherein, the profiled wire is suitable for use as a flexible tube component in offshore oil well drilling and comprising in percent by weight of the total mass,

$$0.75 \leq C \% \leq 0.95;$$

$$0.30 \leq Mn \% \leq 0.85;$$

$$Cr \leq 0.4\%;$$

$$V \leq 0.16\%;$$

$$Si \leq 1.40\%; \text{ and}$$

the remainder being iron and impurities resulting from processing of metal in a liquid state.

2. The process according to claim 1, wherein the Si is $\geq 0.15\%$.
3. The process according to claim 1 or 2, wherein the profiled wire further comprises in percent by weight of the total mass not more than 0.06% Al.
4. The process according to any one of claims 1 to 3, wherein the profiled wire further comprises in percent by weight of the total mass not more than 0.1% Ni.
5. The process according to any one of claims 1 to 4, wherein the profiled wire further comprises in percent by weight of the total mass not more than 0.1% Cu.