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(54) **COLD FORGING WIRE ROD AND STEEL PART HAVING IMPROVED DELAYED FRACTURE RESISTANCE, AND METHOD FOR MANUFACTURING SAME**

(57) Disclosed are: a cold forging wire rod and a steel part in which the microstructure is controlled through the alloy composition and manufacturing method, thus enabling cost reduction and enhancing delayed fracture resistance; and a method for manufacturing same. The steel part having improved delayed fracture resistance according to an embodiment of the present invention contains, in weight percent, 0.18-0.25% of C, 0.30-0.50% of Si, 0.35-0.50% of Mn, more than 0% and no more than 0.03% of P, more than 0% and no more than 0.03% of S, 0.45-0.60% of Cr, 0.015-0.03% of Ti, 0.001-0.004% of B, with the remainder comprising Fe and inevitable impurities, and may include at least 90% by volume of auto-tempered martensite.

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Description

[Technical Field]

[0001] The disclosure relates to a wire rod for cold forging and steel part and method of manufacturing them, and more particularly, to a wire rod for cold forging and steel part and method of manufacturing them, which improves delayed fracture resistance by controlling the microstructure.

[Background Art]

[0002] Wire rods used for fastening bolts for vehicles and structures are required to have high strength of the steel material to lighten the vehicles and miniaturize the structures. To increase the strength of the steel material, metal strengthening mechanisms such as cold working, grain refinement, martensite strengthening, precipitation hardening, etc., are used. However, dislocations, grain boundaries, martensitic lath boundaries, fine precipitate boundaries, etc., used for the strengthening mechanisms act as hydrogen trap sites in the steel material and as causes for deterioration of delayed fracture. Hence, delayed fracture in high-strength bolts with a tensile strength of at least 1 GPa deteriorates.

[0003] To solve this problem, the steel material for bolts of at least 1 GPa having the traditional tempered martensitic structure has used Mo-added Cr-Mo alloy steel. However, in order to respond to the need for cost reduction along with the development of bolt manufacturing process technology, there have been efforts to replace the high-strength steel of at least 1 GPa with boron-added steel. As a result, the boron-added steel is used to realize cost reduction, and after the safety is confirmed, the boron-added steel is being applied to some fastening bolts for vehicles.

[0004] However, when the boron-added steel is used at 1.1 GPa or higher, hydrogen delayed cracking occurs (Ref. N.Uno et al., Nippon Steel Technical Report No.97 (2008)). Accordingly, for high-strength steel of at least 1.1 GPa, MO-added standard steel is applied, or MO- or V-added steel types that are proprietary to the respective steel companies have been used. However, for cost competitiveness, there is a need to develop a high-strength steel that omits expensive carbide elements such as Mo and V.

[Disclosure]

[Technical Problem]

[0005] To solve the aforementioned problem, the disclosure aims to provide a wire rod for cold forging and steel part and method for manufacturing them, which enables cost reduction and improves delayed fracture resistance by controlling the microstructure through an alloy composition and manufacturing method.

[Solution to Problem]

[0006] According to an embodiment of the disclosure, a steel part with improved delayed fracture resistance includes, in percent by weight (wt%), 0.18 to 0.25% of C, 0.30 to 0.50% of Si, 0.35 to 0.50% of Mn, more than 0 to 0.03% of P, more than 0 to 0.03% of S, 0.45 to 0.60% of Cr, 0.015 to 0.03% of Ti, 0.001 to 0.004% of B, the remainder having Fe and other unavoidable impurities, wherein autotempered martensite is at least 90% in volume fraction.

[0007] In an embodiment of the disclosure, the steel part with improved delayed fracture resistance may have carbides in prior austenite grains having an average thickness of 15 nm or less.

[0008] In an embodiment of the disclosure, the steel part with improved delayed fracture resistance may have at least 1200 MPa of tensile strength.

[0009] According to an embodiment of the disclosure, a method of manufacturing a steel part with improved delayed fracture resistance includes preparing a steel material including, in percent by weight (wt%), 0.18 to 0.25% of C, 0.30 to 0.50% of Si, 0.35 to 0.50% of Mn, more than 0 to 0.03% of P, more than 0 to 0.03% of S, 0.45 to 0.60% of Cr, 0.015 to 0.03% of Ti, 0.001 to 0.004% of B, the remainder having Fe and other unavoidable impurities; preparing a wire rod by finish-rolling the steel material; winding the wire rod; drawing and then performing spheroidizing heat treatment on the wound wire rod; shaping the spheroidized heat-treated wire rod into a part; and austenizing and then quenching the part.

[0010] In an embodiment of the disclosure, in the method of manufacturing a steel part with improved delayed fracture resistance, the finish-rolling may be performed at 880 to 980 °C and the winding may be performed at 830 to 930 °C.

[0011] In an embodiment of the disclosure, in the method of manufacturing a steel part with improved delayed fracture resistance, the spheroidizing heat treatment may be performed at a range of 745 to 765 °C of maximum temperature.

[0012] In an embodiment of the disclosure, in the method of manufacturing a steel part with improved delayed fracture resistance, the austenizing may be performed at 870 to 940 °C.

[0013] In an embodiment of the disclosure, in the method of manufacturing a steel part with improved delayed fracture resistance, the quenching may be performed with a refrigerant of 10 to 80 °C.

[0014] According to an embodiment of the disclosure, a wire rod for cold forging includes, in percent by weight (wt%), 0.18 to 0.25% of C, 0.30 to 0.50% of Si, 0.35 to 0.50% of Mn, more than 0 to 0.03% of P, more than 0 to 0.03% of S, 0.45 to 0.60% of Cr, 0.015 to 0.03% of Ti, 0.001 to 0.004% of B, the remainder having Fe and other unavoidable impurities, wherein a diameter of the wire rod is 5.5 to 20 mm.

[Advantageous Effects]

[0015] According to an embodiment of the disclosure, a wire rod for cold forging and steel part and method for manufacturing them, which enables cost reduction and improves delayed fracture resistance by controlling the microstructure through an alloy composition and manufacturing method may be provided.

[Best Mode]

[0016] According to an embodiment of the disclosure, a steel part with improved delayed fracture resistance includes, in percent by weight (wt%), 0.18 to 0.25% of C, 0.30 to 0.50% of Si, 0.35 to 0.50% of Mn, more than 0 to 0.03% of P, more than 0 to 0.03% of S, 0.45 to 0.60% of Cr, 0.015 to 0.03% of Ti, 0.001 to 0.004% of B, the remainder having Fe and other unavoidable impurities, wherein autotempered martensite is at least 90% in volume fraction.

[Modes]

[0017] The following embodiments are provided as examples to convey the full spirit of the disclosure to those of ordinary skill in the art to which the embodiments of the disclosure belong. The disclosure is not limited to the embodiments but may be specified in any other forms. In the drawings, unrelated part of the description is not shown to clarify the disclosure, and the size of an element may be a little exaggerated to help understanding.

[0018] Throughout the specification, the term "include (or including)" or "comprise (or comprising)" is inclusive or open-ended and does not exclude additional, unrecited components, elements or method steps, unless otherwise stated.

[0019] It is to be understood that the singular forms "a," "an," and "the" include plural references unless the context clearly dictates otherwise.

[0020] A reason for numerical limitation of the content of an alloy composition in an embodiment of the disclosure will now be described. A unit of weight(wt)% will now be used unless otherwise mentioned.

[0021] According to an embodiment of the disclosure, a steel part with improved delayed fracture resistance includes, in percent by weight (wt%), 0.18 to 0.25% of C, 0.30 to 0.50% of Si, 0.35 to 0.50% of Mn, more than 0 to 0.03% of P, more than 0 to 0.03% of S, 0.45 to 0.60% of Cr, 0.015 to 0.03% of Ti, 0.001 to 0.004% of B, the remainder having Fe and other unavoidable impurities.

[0022] The content of C (carbon) may be 0.18 to 0.25%.

[0023] C is an element added to secure strength of the product. Considering this, at least 0.18% of C may be added. However, when the content of C is excessive, it may cause delayed fracture due to the rise in strength. Considering this, the upper limit of the content of C is limited to 0.25%.

[0024] The content of Si (silicon) may be 0.30 to 0.50%.

[0025] Si may be added for deoxidation of steel. Furthermore, Si is an element that is effective in securing strength through solid solution strengthening. Considering this, Si may be added in at least 0.30%. However, when the content of Si is excessive, impact characteristics and formability may deteriorate. Considering this, the upper limit of the content of Si may be limited to 0.50%. Preferably, the content of Si may be 0.31 to 0.48%.

[0026] The content of Mn (manganese) may be 0.35 to 0.50%.

[0027] Mn is an element that is very effective in enhancing hardenability and producing a solid solution strengthening effect by forming a substitutional solid solution within the matrix structure. When the content of Mn is low, it may not sufficiently combine with S (sulfur) brought as an impurity in the steel, thereby causing casting cracks. Considering this, at least 0.35% of Mn may be added. However, when the content of Mn is excessive, it may form MnS, which may deteriorate delayed fracture resistance. Considering this, the upper limit of the content of Mn may be limited to 0.50%. Preferably, the content of Mn may be 0.36 to 0.49%.

[0028] The content of P (phosphorus) may be more than 0 to 0.03%.

[0029] P is segregated on the grain boundaries, reducing toughness, and acts as a cause to reduce delayed fracture resistance. Hence, it may be managed as an impurity in the disclosure. Considering this, the upper limit of the P content may be limited to 0.03%, and is desirable to be as close as 0%.

[0030] The content of S (sulfur) may be more than 0 to 0.03%.

[0031] Like P, S is segregated on the grain boundaries, reducing toughness, and acts as a cause to hinder hot rolling by forming low melding point sulfides. Hence, it may be managed as an impurity in the disclosure. Considering this, the upper limit of the S content may be limited to 0.03%, and is desirable to be as close as 0%.

[0032] The content of Cr (chrome) may be 0.45 to 0.60%.

[0033] Cr is an element that is very effective in enhancing hardenability and producing a solid solution strengthening effect by forming a substitutional solid solution within the matrix structure. Considering this, Cr may be added in at least 0.45%. However, when the Cr content is excessive, the c/a ratio of corrosion pits increases due to formation of a chromium oxide layer on the surface, so the delayed fracture resistance may deteriorate due to the notch effect. Considering this, the upper limit of the content of Cr may be limited to 0.60%. Preferably, the content of Cr may be 0.46 to 0.59%.

[0034] The content of Ti (titanium) may be 0.015 to 0.03%.

[0035] Ti is an effective element in preventing B (boron) from combining with N (nitrogen) by combining with N brought into the steel into a titanium nitride. Considering this, Ti may be added in at least 0.015%. However, when the content of Ti is excessive, it may form coarse carbonitrides, which may deteriorate delayed fracture resistance. Considering this, the upper limit of the content of Ti may be limited to 0.03%. Preferably, the content of Ti may be 0.023 to 0.026%.

[0036] The content of B (boron) may be 0.001 to 0.004%.

[0037] B is an effective element in improving hardenability. Considering this, B may be added in at least 0.001%. However, when the content of B is excessive, it may form $Fe_{23}(CB)_6$ carbides, which may cause brittleness of the austenite grain boundaries and thus, deteriorate delayed fracture resistance. Considering this, the upper limit of the content of B may be limited to 0.004%. Preferably, the content of B may be 0.0018% to 0.0023%.

[0038] The remaining ingredient is iron (Fe) in the disclosure. However, unintended impurities may be inevitably mixed in from raw materials or surroundings in the normal manufacturing process, so they may not be excluded. These impurities may be known to anyone skilled in the ordinary manufacturing process, so not all of them are specifically mentioned in this specification.

[0039] In an embodiment of the disclosure, a steel part with improved delayed fracture resistance may have at least 90% of autotempered martensite in volume fraction.

[0040] When the autotempered martensite is less than 90%, it is difficult to secure sufficient toughness, which may deteriorate delayed fracture resistance. Hence, in the disclosure, the autotempered martensite may be controlled to be at least 90% through an alloy composition and manufacturing process. Especially, the autotempered martensite structure is characterized by being automatically tempered during quenching without an extra tempering heat treatment process.

[0041] Furthermore, a steel part with improved delayed fracture resistance according to an embodiment of the disclosure may be automatically tempered during quenching without an extra tempering heat treatment process, so that the average thickness of carbides in prior austenitic grains may be controlled to be 15 nm or less.

[0042] It is known that the surface direction of the carbides precipitated in a plate type during autotempering has high coherency and is thus ineffective as a hydrogen trap site, and that the plate type side surface has low coherency, acting as a non-diffusive hydrogen trap site and thus improves hydrogen delayed fracture resistance.

[0043] Hence, when the average thickness of carbides in the prior austenite grains is thin, the coherency of the carbide interface increases, making it difficult to improve hydrogen delayed fracture resistance. On the other hand, when the average thickness of carbides in the prior austenite grains is thick, the number of carbides decreases, making it difficult to improve delayed fracture resistance. Hence, in the disclosure, the average thickness of the carbides in prior austenite grains is controlled to be 15 nm or less to improve the delayed fracture resistance.

[0044] In an embodiment of the disclosure, the steel part with improved delayed fracture resistance may have a tensile strength of at least 1200 MPa by controlling an alloy composition and manufacturing process.

[0045] Next, a method of manufacturing a steel part with improved delayed fracture resistance according to another aspect of the disclosure will now be described.

[0046] In an embodiment of the disclosure, a method of manufacturing a steel part with improved delayed fracture resistance includes preparing a steel material including, in percent by weight (wt%), 0.18 to 0.25% of C, 0.30 to 0.50% of Si, 0.35 to 0.50% of Mn, more than 0 to 0.03% of P, more than 0 to 0.03% of S, 0.45 to 0.60% of Cr, 0.005 to 0.03% of Ti, 0.001 to 0.004% of B, the remainder having Fe and other unavoidable impurities; preparing a wire rod by finish-rolling the steel material; winding the wire rod; drawing and then performing spheroidizing heat treatment on the wound wire rod; shaping the spheroidized heat-treated wire rod into a part; austenizing and then quenching the part.

[0047] The reason of numerical limitations of ranges of ingredients of each alloy composition is the same as described above, and each manufacturing step will now be described in more detail.

[0048] After the steel material that satisfies the alloy composition is prepared first, it may go through a series of finish-rolling, winding, spheroidizing heat treatment, shaping, austenizing and quenching processes.

[0049] First, a wire rod may be prepared by finish-rolling the steel material at 880 to 980 °C, and the wire rod may be wound at 830 to 930 °C.

[0050] When the finish-rolling temperature or the winding temperature is low, the surface layer is a quasi-two-phase station, so a surface ferrite decarburization layer may be formed by phase transformation. Accordingly, when the finish-rolling temperature or the winding temperature is low, the ferrite decarburization layer is formed on the surface even during heat treatment of the steel part, deteriorating the delayed fracture resistance. With consideration for this, the finish-rolling temperature may be at least 880 °C or the winding temperature may be at least 830 °C.

[0051] On the other hand, when the finish-rolling temperature or the winding temperature is high, the ferrite decarburization layer is formed on the surface as diffusion accelerates the decarburization, thereby deteriorating the delayed fracture resistance. With consideration for this, the finish-rolling temperature may be 980 °C or less, or the winding temperature may be 930 °C or less.

[0052] Subsequently, the wound wire rod may be drawn to be fit for the purpose, and subjected to spheroidizing heat treatment at a range of 745 to 765 °C of maximum temperature.

[0053] When the maximum temperature for the spheroidizing heat treatment is too low or too high, the hardness of a material to be subject to the spheroidizing heat treatment increases, which may cause cracks due to deterioration of formability when the steel part is processed. Considering this, the maximum temperature at which to perform the spheroidizing heat treatment may be 745 to 765 °C.

[0054] The spheroidized heat treated wire rod is shaped to be fit for the purpose into a steel part, and the steel part may be austenized at 870 to 940 °C.

[0055] When the austenizing temperature is low, austenite reverse transformation does not occur sufficiently, so the toughness may deteriorate because of unevenness of the martensite structure after quenching. Considering this, the austenizing temperature may be at least 870 °C. On the other hand, when the austenizing temperature is high, the austenite grain size becomes coarse, which may deteriorate the delayed fracture resistance. Considering this, the upper limit of the austenizing temperature may be limited to 940 °C.

[0056] Subsequently, the austenized steel part may be quenched with a refrigerant of 10 to 80 °C.

[0057] When the temperature of the quenching refrigerant is low, fine quenching cracks are formed due to heat deformation of the steel part, causing delayed destruction. Considering this, the temperature of the quenching refrigerant may be at least 10 °C. On the other hand, when the temperature of the quenching refrigerant is high, the autotempering effect may increase, making it difficult to realize a targeted strength. Considering this, the upper limit of the temperature of the quenching refrigerant may be limited to 80 °C.

[0058] From the aforementioned process, a final microstructure of the steel part may realize at least 90% of autotempered martensitic structure without a tempering process and realize a structure in which carbides with an average thickness of 15 nm or less within the prior austenite grains are precipitated. Accordingly, the delayed fracture resistance may be improved by controlling the microstructure.

[0059] Next, a wire rod for cold forging according to another aspect of the disclosure will now be described.

[0060] According to an embodiment of the disclosure, a wire rod for cold forging includes, in percent by weight (wt%), 0.18 to 0.25% of C, 0.30 to 0.50% of Si, 0.35 to 0.50% of Mn, more than 0 to 0.03% of P, more than 0 to 0.03% of S, 0.45 to 0.60% of Cr, 0.005 to 0.03% of Ti, 0.001 to 0.004% of B, the remainder having Fe and other unavoidable impurities, wherein the wire rod has a diameter of 5.5 to 20 mm.

[0061] The reason of limiting the numerical values of each ingredient range of the alloy composition is as described above, and the wire rod for cold forging according to an embodiment of the disclosure may be manufactured to have a diameter of 5.5 to 20 mm. It is not, however, limited thereto, and may be manufactured to have various diameters for the purposes.

[0062] Embodiments of the disclosure will now be described in more detail. The embodiments may be merely for illustration, and the disclosure is not limited thereto. The scope of the disclosure is defined by the claims and their equivalents.

[0063] {Embodiment}

[0064] With various alloy ingredient ranges shown in table 1 below, a steel material was manufactured, a wire rod having a diameter of 15 mm is prepared by finish-rolling the steel material at 910 °C and then wound at 880 °C into a coil form. The wound wire rod may be subjected to spheroidizing heat treatment at a range having a maximum temperature of 755 °C, shaped into bolts of a screw-M12 standard, austenized at 890 °C, and quenched with a refrigerant of 60 °C. The spheroidizing heat treatment temperature refers to the highest heating temperature.

[Table 1]

section	alloy ingredients							
	C	Si	Mn	P	S	Cr	Ti	B
Embodiment 1	0.18	0.40	0.46	0.011	0.005	0.53	0.024	0.001 8
Embodiment 2	0.25	0.41	0.45	0.010	0.005	0.54	0.024	0.002 1
Embodiment 3	0.20	0.40	0.36	0.009	0.005	0.52	0.026	0.002 3
Embodiment 4	0.21	0.44	0.49	0.011	0.005	0.52	0.023	0.002 1

(continued)

section	alloy ingredients							
	C	Si	Mn	P	S	Cr	Ti	B
Embodiment 5	0.19	0.41	0.44	0.011	0.005	0.46	0.024	0.002 0
Embodiment 6	0.22	0.41	0.43	0.010	0.005	0.59	0.025	0.002 1
Embodiment 7	0.23	0.31	0.42	0.009	0.005	0.52	0.024	0.002 2
Embodiment 8	0.21	0.48	0.44	0.010	0.005	0.53	0.024	0.001 8
Comparative example 1	0.17	0.41	0.46	0.011	0.005	0.52	0.024	0.002 1
Comparative example 2	0.26	0.42	0.47	0.010	0.005	0.52	0.023	0.002 3
Comparative example 3	0.18	0.25	0.45	0.011	0.005	0.50	0.024	0.002 2
Comparative example 4	0.24	0.40	0.53	0.011	0.005	0.51	0.024	0.002 0
Comparative example 5	0.23	0.37	0.49	0.009	0.005	0.42	0.023	0.001 9
Comparative example 6	0.22	0.42	0.47	0.009	0.005	0.64	0.022	0.002 1

[0065] Table 2 below shows tensile strength, carbide thickness and crack presence from delayed fracture performance evaluation of the manufactured bolt. The tensile strength was measured by ZWICK Z250 tensile tester from Zwick/Roell. The tensile strength measurement test was performed with a tensile specimen having a diameter of 10 mm and a gauge diameter of 6.25 mm.

[0066] The carbide thickness measurement was performed with the FEI Tecnai OSIRIS transmission electron microscopy (TEM). In this case, the carbide thickness was measured at 5 random points on a replica specimen and expressed as the average thickness, and the minor axis of the carbide formed in a plate type was defined and measured as the thickness.

[0067] The delayed fracture performance evaluation test was performed in a delayed fracture simulation method by which the bolt is fastened to a structure with a clamping force of yield strength and presence or absence of a crack on the screw thread, which is a stress-concentrated area, is observed before/after the bolt fastened to the structure is immersed in a 5% hydrochloric acid + 95% distilled solution for 10 minutes.

[0068] As a result of the delayed fracture performance evaluation, when there is a crack, it is marked by 'O', or when there is no crack, it is marked by 'X'.

[Table 2]

section	tensile strength (MPa)	carbide thickness (mn)	presence of crack
Embodiment 1	1213	12	X
Embodiment 2	1680	13	X
Embodiment 3	1365	11	X
Embodiment 4	1391	11	X
Embodiment 5	1265	12	X
Embodiment 6	1354	13	X
Embodiment 7	1566	14	X
Embodiment 8	1421	10	X
Comparative example 1	**1185**	13	X
Comparative example 2	1693	**16**	**O**
Comparative example 3	**1193**	**16**	**O**
Comparative example 4	1655	14	**O**
Comparative example 5	1586	13	**O**

(continued)

section	tensile strength (MPa)	carbide thickness (mn)	presence of crack
Comparative example 6	1543	15	**O**

[0069] Referring to table 2, embodiments 1 to 8 satisfied the alloy composition, ingredient ranges and manufacturing processes proposed in the disclosure. Accordingly, embodiments 1 to 8 satisfied a tensile strength of at least 1200 MPa and a carbide thickness of 15 nm or less, and resulted in no cracks as a result of the delayed fracture performance evaluation. On the other hand, comparative example 1 failed to satisfy the tensile strength of 1200 MPa because the C content was low.

[0070] In comparative example 2, the carbide thickness exceeded 15 nm because of the high C content, and cracks occurred as a result of the delayed fracture performance evaluation.

[0071] Comparative example 3 failed to satisfy the tensile strength of 1200 MPa because the Si content was low.

[0072] Comparative example 4 had the high Mn content and formed coarse MnS, which caused occurrence of cracks as a result of the delayed fracture performance evaluation.

[0073] In comparative example 5, the Cr content was low and a bainite mixed structure was formed in the microstructure, resulting in occurrence of cracks as a result of the delayed fracture performance evaluation.

[0074] Comparative example 6 had a high Cr content, forming sharp corrosion pits when corroded by hydrochloric acid, and thus causing occurrence of cracks as a result of the delayed fracture performance evaluation.

[0075] Next, a steel material was manufactured with the alloy ingredients of embodiment 5 in table 1, and manufactured into a bolt at finish-rolling temperature, winding temperature, maximum temperature of spheroidizing heat treatment and austenizing temperature as shown in table 3 below, and then presence or absence of cracks is marked in table 3 as a result of delayed fracture performance evaluation.

[Table 3]

section	temperature (°C)				presence of crack
	finish-rolling	winding	spheroidizing heat treatment	austenizing	
Embodiment 5	910	880	755	890	X
Comparative example 7	**985**	**935**	755	890	O
Comparative example 8	**865**	**825**	755	890	O
Comparative example 9	910	880	755	**950**	O
Comparative example 10	910	880	755	**860**	O
Comparative example 11	910	880	**740**	890	O
Comparative example 12	910	880	**770**	890	O

[0076] Comparative example 7 had high finish-rolling temperature and winding temperature, which caused the prior austenite grain size to grow, resulting in occurrence of cracks as a result of delayed fracture performance evaluation. Comparative example 8 had low finish-rolling temperature and winding temperature, which caused formation of ferrite decarburization layer on the wire rod, resulting in occurrence of cracks as a result of delayed fracture performance evaluation.

[0077] Comparative example 9 had high austenizing temperature, which caused the prior austenite grain size to grow, resulting in occurrence of cracks as a result of delayed fracture performance evaluation.

[0078] Comparative example 10 had low austenizing temperature, making it enter into the quasi-two-phase station, forming a ferrite decarburization layer during heating, and resulting in occurrence of cracks as a result of delayed fracture performance evaluation.

[0079] Comparative examples 11 and 12 had low and high maximum temperatures for spheroidizing heat treatment, respectively, so the spheroidizing heat treatment was not sufficiently done, leading to deterioration of formability. Accordingly, comparative examples 11 and 12 had cracks formed during screw part shaping, resulting in occurrence of cracks as a result of delayed fracture performance evaluation.

[Table 4]

	austenizi ng temperat ure (°C)	quenching oil temperature (°C)	automartens ite fraction (%)	carbide thickness (nm)	delayed fracture crack presence
Embodiment 5	910	60	95%	12	X
Comparative example 13	910	85	**89%**	**16**	**O**
Comparative example 14	910	95	**86%**	**18**	**O**
Comparative example 15	910	105	**85%**	**21**	**O**
Comparative example 16	910	115	**85%**	**22**	**O**

[0080] Comparative examples 13 to 16 used a steel material having the same alloy ingredients and composition ranges as in embodiment 5, but as compared to embodiment 5, they had an automartensite fraction and carbide thickness that did not satisfy the ranges of the disclosure, which caused occurrence of cracks as a result of delayed fracture performance evaluation.

[Industrial Applicability]

[0081] According to the disclosure, a wire rod for cold forging and steel part and method for manufacturing them, which enables cost reduction and improves delayed fracture resistance by controlling the microstructure through an alloy composition and manufacturing method may be provided, so the industrial applicability is acknowledged.

Claims

1. A steel part with improved delayed fracture resistance comprising:

in percent by weight (wt%), 0.18 to 0.25% of C, 0.30 to 0.50% of Si, 0.35 to 0.50% of Mn, more than 0 to 0.03% of P, more than 0 to 0.03% of S, 0.45 to 0.60% of Cr, 0.015 to 0.03% of Ti, 0.001 to 0.004% of B, the remainder having Fe and other unavoidable,
wherein autotempered martensite is at least 90% in volume fraction.

2. The steel part of claim 1, wherein carbides in prior austenite grains have an average thickness of 15 nm or less.

3. The steel part of claim 1, wherein tensile strength is at least 1200 MPa.

4. A method of manufacturing a steel part with improved delayed fracture resistance, the method comprising:

preparing a steel material including, in percent by weight (wt%), 0.18 to 0.25% of C, 0.30 to 0.50% of Si, 0.35 to 0.50% of Mn, more than 0 to 0.03% of P, more than 0 to 0.03% of S, 0.45 to 0.60% of Cr, 0.015 to 0.03% of Ti, 0.001 to 0.004% of B, the remainder having Fe and other unavoidable;
preparing a wire rod by finish-rolling the steel material;
winding the wire rod;
drawing and then performing spheroidizing heat treatment on the wound wire rod;
shaping the spheroidized heat-treated wire rod into a part; and
austenizing and then quenching the part.

5. The method of claim 4, wherein:

the finish-rolling is performed at 880 to 980 °C, and
wherein the winding is performed at 830 to 930 °C.

6. The method of claim 4, wherein the spheroidizing heat treatment is performed at a range of 745 to 765 °C of maximum temperature.

7. The method of claim 4, wherein the austenizing is performed at 870 to 940 °C.

8. The method of claim 4, wherein the quenching is performed with a refrigerant of 10 to 80 °C.

9. A wire rod for cold forging comprising:

in percent by weight (wt%), 0.18 to 0.25% of C, 0.30 to 0.50% of Si, 0.35 to 0.50% of Mn, more than 0 to 0.03% of P, more than 0 to 0.03% of S, 0.45 to 0.60% of Cr, 0.015 to 0.03% of Ti, 0.001 to 0.004% of B, the remainder having Fe and other unavoidable,
wherein the wire rod has a diameter of 5.5 to 20 mm.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/KR2022/019409

A. CLASSIFICATION OF SUBJECT MATTER

C22C 38/28(2006.01)i; **C22C 38/32**(2006.01)i; **B21B 1/16**(2006.01)i; **C21D 9/52**(2006.01)i; **C21D 1/18**(2006.01)i; **B21C 47/02**(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C22C 38/28(2006.01); B21J 5/00(2006.01); C21D 1/06(2006.01); C21D 1/19(2006.01); C21D 8/00(2006.01); C21D 8/06(2006.01); C21D 9/46(2006.01); C22C 38/00(2006.01); C22C 38/22(2006.01); C22C 38/32(2006.01); C22C 38/60(2006.01)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Korean utility models and applications for utility models: IPC as above
Japanese utility models and applications for utility models: IPC as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

eKOMPASS (KIPO internal) & keywords: 냉간단조(cold forging), 선재(wire rod), 내지연파괴저항성(delayed fracture resistance), 오토템퍼드 마르텐사이트(autotempered martensite), 구상화열처리(spheroidizing)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	KR 10-1322534 B1 (NIPPON STEEL CORPORATION) 28 October 2013 (2013-10-28) See paragraphs [0122], [0124], [0132]-[0133] and [0139], claims 1, 3 and 6 and table 2.	1,3,9 2,4-8
Y	KR 10-2021-0078116 A (POSCO) 28 June 2021 (2021-06-28) See paragraphs [0057], [0061]-[0062] and [0064].	2,4-8
A	KR 10-1225404 B1 (JFE STEEL CORPORATION) 22 January 2013 (2013-01-22) See paragraphs [0056]-[0057] and claim 1.	1-9
A	KR 10-2014-0123111 A (KABUSHIKI KAISHA KOBE SEIKO SHO (KOBE STEEL, LTD.)) 21 October 2014 (2014-10-21) See paragraphs [0061]-[0062] and claim 1.	1-9

☑ Further documents are listed in the continuation of Box C. ☑ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"D" document cited by the applicant in the international application
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"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
10 March 2023	**10 March 2023**
Name and mailing address of the ISA/KR **Korean Intellectual Property Office** **Government Complex-Daejeon Building 4, 189 Cheongsa-ro, Seo-gu, Daejeon 35208** Facsimile No. **+82-42-481-8578**	Authorized officer Telephone No.

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/KR2022/019409

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 11-236617 A (KOBE STEEL LTD.) 31 August 1999 (1999-08-31) See paragraphs [0026]-[0031] and claims 1-2.	1-9

Form PCT/ISA/210 (second sheet) (July 2022)

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/KR2022/019409

Patent document cited in search report	Publication date (day/month/year)	Patent family member(s)	Publication date (day/month/year)
KR 10-1322534 B1	28 October 2013	CN 102791898 A	21 November 2012
		EP 2546380 A1	16 January 2013
		EP 2546380 B1	08 June 2016
		ES 2583053 T3	16 September 2016
		JP 5135557 B2	06 February 2013
		JP WO2011-111873 A1	27 June 2013
		PL 2546380 T3	30 November 2016
		US 2012-0298262 A1	29 November 2012
		US 8951365 B2	10 February 2015
		WO 2011-111873 A1	15 September 2011
KR 10-2021-0078116 A	28 June 2021	CN 114929922 A	19 August 2022
		EP 4060072 A1	21 September 2022
		US 2023-0020467 A1	19 January 2023
		WO 2021-125555 A1	24 June 2021
KR 10-1225404 B1	22 January 2013	CA 2713195 A1	06 August 2009
		CA 2713195 C	26 November 2013
		CN 101932745 A	29 December 2010
		CN 101932745 B	08 August 2012
		EP 2258887 A1	08 December 2010
		EP 2258887 B1	29 July 2015
		JP 2009-203549 A	10 September 2009
		JP 5365216 B2	11 December 2013
		MX 2010008404 A	25 October 2010
		US 2011-0048589 A1	03 March 2011
		US 8840834 B2	23 September 2014
		WO 2009-096595 A1	06 August 2009
KR 10-2014-0123111 A	21 October 2014	CA 2864453 A1	03 October 2013
		CA 2864453 C	03 November 2015
		CN 104204254 A	10 December 2014
		CN 104204254 B	22 June 2016
		EP 2832875 A1	04 February 2015
		JP 2013-227647 A	07 November 2013
		JP 6034632 B2	30 November 2016
		MX 2014011470 A	08 December 2014
		TW 201348460 A	01 December 2013
		TW I484045 B	11 May 2015
		US 2015-0053315 A1	26 February 2015
		US 9845519 B2	19 December 2017
		WO 2013-145868 A1	03 October 2013
JP 11-236617 A	31 August 1999	JP 3343505 B2	11 November 2002

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REFERENCES CITED IN THE DESCRIPTION

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Non-patent literature cited in the description

- **N.UNO et al.** *Nippon Steel Technical Report,* 2008 **[0004]**