(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
11 June 2020 (11.06.2020)

WIPO | PCT

(10) International Publication Number
WO 2020/116390 A1

(51) International Patent Classification:
C08K 3/04 (2006.01) *F16C 33/00* (2006.01)
C08K 3/08 (2006.01) *B32B 27/08* (2006.01)
C08K 3/30 (2006.01) *C08J 5/18* (2006.01)

(21) International Application Number:
PCT/JP2019/047025

(22) International Filing Date:
02 December 2019 (02.12.2019)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
2018-226541 03 December 2018 (03.12.2018) JP

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(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DJ, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JO, KE, KG, KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, 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) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (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, KM, ML, MR, NE, SN, TD, TG).

Published:
— *with international search report (Art. 21(3))*

(54) **Title:** COMPOSITION, FILM FORMED FROM THE COMPOSITION, SLIDING MEMBER HAVING THE FILM, AND METHOD FOR PRODUCING THE SAME

(57) **Abstract:** A film is formed by use of a composition containing (A) a binder resin, (B) a hard particle, and (C) a solid lubricant, wherein the hard particle contains metal tungsten particle with 0.5 to 10 micrometers of average particle size measured by laser diffraction scattering method.

WO 2020/116390 A1

Description

Title of Invention: COMPOSITION, FILM FORMED FROM THE COMPOSITION, SLIDING MEMBER HAVING THE FILM, AND METHOD FOR PRODUCING THE SAME

Technical Field

[0001] The present invention relates to a composition that provides a lubricating film having excellent sliding property. Furthermore, the present invention relates to a film formed from the composition, a sliding member having the film, and a method of manufacturing the same.

Background Art

[0002] Film-forming compositions to improve the sliding properties of component surfaces are used in various applications such as industrial machines, construction machines and automobiles. Normally, film-forming compositions contain binder and solid lubricants. Film-forming compositions comprising hard particles in addition to binder and solid lubricants are also known. For example, JP2017-201165A discloses a film-forming composition comprising a thermosetting resin, a solid lubricant and/or hard particles, solvent and polyether-modified silicone. However, it discloses metal oxides such as silicon oxide, aluminum oxide, chromium oxide, titanium oxide, zinc oxide, ferric oxide and mullite; metal carbonates such as silicon carbide and metal nitrides such as silicon nitride. JP2000-265953A discloses a compressing machine coated by a film formed from a polymer composition comprising metal particles. It discloses tin, silver, aluminum, copper, zinc, nickel, titanium, tungsten, molybdenum, magnesium and iron and alloy comprising thereof as examples of metal particles. It also discloses the particle size of the metal is preferably from 10 to 100 micron.

[0003] Normally, a sliding member is used to slide two or more components in contact. Sliding member needs to have not only the property for excellent sliding (i.e. less coefficient of friction), but also the property for wear resistance (less abrasion). However, the present inventors have found that when a conventional film comprises metal oxides, metal carbonates or metal nitrides as hard particles, the film tends to abrade the surface of the corresponding materials.

Citation List

Patent Literature

[0004] [PTL1] JP2017-201165A

[PTL2] JP2000-265953A

Summary of Invention

[0005] An object of the present invention is to provide a composition which forms a film having excellent sliding property and the property with less abrasion for sliding member and corresponding material. The present inventors have created the invention based on the finding that a film comprising metal tungsten particles within a specific average particle size can prevent the abrasion for sliding member and corresponding material in the un-expected level.

[0006] The first aspect of the invention relates to a composition comprising (A) a binder resin; (B) a hard particle and (C) a solid lubricant, wherein the hard particle comprises metal tungsten particles with 0.5 to 10 micrometers of average particle size measured by laser diffraction scattering method.

[0007] The second aspect of the invention relates to a lubricating film made from the above composition. In concretely, the second aspect of the invention relates to a film comprising (A) a binder resin; (B) a hard particle and (C) a solid lubricant, wherein the hard particle comprises metal tungsten particles with 0.5 to 10 micrometers of average particle size measured by laser diffraction scattering method.

[0008] The third aspect of the invention relates to a sliding member having the above film.

[0009] The forth aspect of the invention relates to a method for forming a lubricating film on surface of a member, comprising the steps of: (I) preparing a composition comprising (A) a binder resin, (B) a hard particle and (C) a solid lubricant, wherein the hard particle comprises metal tungsten particles with 0.5 to 10 micrometers of average particle size measured by laser diffraction scattering method, (II) applying the composition on surface of a member; and (III) heat-curing the applied composition, thereby forming a lubricating film on surface of the member.

[0010] According to the composition of the present invention, it is possible to form a film having both excellent sliding property and good wear resistances for sliding member and corresponding material.

Description of Embodiments

[0011] The composition used in the present invention comprises (A) a binder resin; (B) a hard particle and (C) a solid lubricant, wherein the hard particle comprises metal tungsten particles with 0.5 to 10 micrometers of average particle size. The average particle size of the hard particle is measured by laser diffraction scattering method. The composition can further comprise (D) a solvent and/or (E) other additives.

[0012] (A) Binder resin

The binder resin used in the present invention is a resin which forms a lubricating film as a heat resistant resin and has a function as a binder for supporting a solid lubricant described later. Binder resins which can be used include, for example, polyamideimide, polyimide, epoxy resin, phenol resin, polyamide, polybenzimidazole,

polyphenyl sulfonate and polyether ether ketone, and one or more of these may be contained. Preferably, the binder resin is thermosetting resin. Preferably, the binder resin contains polyamideimide or polyimide. More preferably, the binder resin contains polyamideimide.

[0013] The content of the binder resin is 10 to 40% by weight, preferably 20 to 30% by weight, based on the weight of the whole composition.

[0014] (B) Hard particles

The hard particles used in the present invention comprises metal tungsten particles with 0.5 to 10 micrometers of average particle size. The average particle size can be measured by laser diffraction scattering method. Preferably, the average particle size of the metal tungsten particles is from 1 to 6 micrometers, more preferably from 1 to 3 micrometers. The hard particles have the function of improving the load resistance of the film and improving the wear resistance. However, the hard particles sometimes abrade the member located corresponding side of the film. It is un-expected thing that when metal tungsten particles with 0.5 to 10 micrometers of average particle size are used as a hard particle, the abrasion of surface of the corresponding member is low thus the lifetime of the sliding member would be extended.

[0015] Preferable metal tungsten particles are monodispersed fine metal tungsten particles that almost all of the particle is existing as primary particles with less cohesion, or uniform fine metal tungsten particles that have uniform figure of tungsten particles although there are secondary particles. The most preferable metal tungsten particles are monodispersed fine metal tungsten particles.

[0016] The hard particle of the invention can further comprise other hard particles in addition to metal tungsten particles in the amount as long as the function of the composition is not impaired. Other hard particles comprise tungsten carbide, titanium carbide, zirconium carbide, zirconium oxide, tungsten disulfide, molybdenum carbide, tungsten disilicide, titanium nitride and zirconium nitride, and one or more of there. When the other hard particles are contained in addition to metal tungsten particles, the content of metal tungsten particles in the whole hard particles is preferably 50% by weight or more, more preferably 70% by weight or more based on the total weight of the hard particles.

[0017] The content of the hard particles depends on the compounding amount of the solid lubricant described later, but as an example, it is 70 to 140 parts by weight with respect to 100 parts by weight of the binder resin.

[0018] (C) Solid lubricant

The solid lubricant used in the present invention is molybdenum disulfide or graphite. The solid lubricant has a function to improve the sliding property of the film. In particular, molybdenum disulfide and graphite are used in the composition of the

present invention from the viewpoint of forming a film having the sliding property of good wear resistance. Among the two, graphite is particularly preferred in view of lowering the coefficient of friction and further imparting abrasion resistance.

[0019] The content of the solid lubricant depends on the blending amount of the hard particles. Exemplarily, the composition of the present invention comprises 40 to 70 parts by weight of solid lubricant with respect to 100 parts by weight of the binder resin.

[0020] The average particle size of the solid lubricant of the present invention can be measured by laser diffraction scattering method. The average particle diameter of the solid lubricant is preferably 0.1 to 15.0 micrometers, and particularly preferably 1.0 to 10.0 micrometers.

[0021] (D) Solvent

The composition of the present invention can further contain a solvent for the purpose of improving coating properties and the like. The solvent can be selected depending on the type of binder resin. Usable solvents include, for example, ketones such as acetone, methyl ethyl ketone, methyl isobutyl ketone and cyclohexanone; esters such as methyl acetate, ethyl acetate and γ-butyrolactone (GBL); aromatic hydrocarbons such as toluene and xylene; organic halogen compounds such as chloroform, trichloroethylene and trichlorotrifluoroethane; amides such as N-methyl-2-pyrrolidone (NMP), N-ethyl-2-pyrrolidone (NEP), 1,3-Dimethyl-2-imidazolidinone (DMI), 3-methoxy-N,N-dimethylpropanamide, 1-butyl-2-pyrrolidone, N,N-dimethylformamide (DMF) and N,N-dimethylacetoamide (DMAC). The solvent of the present invention may be one or mixed of two or more. Particularly preferred solvents contain NEP, DMI, GBL and 3-methoxy-N,N-dimethylpropanamide.

[0022] (E) Other additives

The coating composition of the present invention may further contain, if necessary, one or more of other additives as long as the object of the present invention is not impaired. Usable additives include, a UV absorber, a light stabilizer, an antioxidant, a thermal polymerization inhibitor, a leveling agent, a deformer, a thickener, an anti-settling agent, pigments (organic colored pigments, inorganic pigments), colored dyes, infrared absorbers, fluorescent whitening agents, dispersants, conductive fine particles, antistatic agents, antifogging agents and coupling agents.

[0023] The composition of the present invention can be produced by appropriately mixing the components (A) to (C) and optionally the components (D) and (E) described above.

[0024] The second aspect of the present invention relates to a film comprising (A) a binder resin; (B) a hard particle and (C) a solid lubricant, wherein the hard particle comprises metal tungsten particles with 0.5 to 10 micrometers of average particle size measured

by laser diffraction scattering method. The film is useful for a lubricating film of a sliding member. The film is formed by the composition of the above mentioned first aspect of the invention. The film of the present invention is a sliding film in which (B) hard particles and (C) a solid lubricant are dispersed in (A) a binder resin. The film can further contain (E) other additives. The film thickness is 1 to 50 μm, preferably 5 to 30 μm.

[0025] The third aspect of the present invention relates to a sliding member having a film of above mentioned the second aspect of the invention. The film is formed on a sliding surface of a sliding member, and it works as a lubricating film. The sliding member includes, for example, a swash plate of a compressor, an engine tappet (valve lifter), a camshaft, a crankshaft, an engine metal, an engine piston, a piston ring, a gear, a door lock, a brake shim or a brake clip.

[0026] The fourth aspect of the present invention is a method of forming a lubricating film on a surface of a member, and it includes the following steps (I) to (III).

[0027] Step (I)

Step (I) is a step of preparing a composition containing (A) a binder resin, (B) a hard particle, and (C) a solid lubricant, in which the hard particle comprises metal tungsten particles with 0.5 to 10 micrometers of average particle size measured by laser diffraction scattering method. The composition is as described above as the first aspect of the present invention. The step can be conducted by mixing components (A) to (E).

[0028] Step (II)

Step (II) is a step of applying the composition prepared in step (I) onto the surface of a member. The composition may be applied by dipping, spin coating, flow coating, spraying, bar coating, gravure coating, roll coating, blade coating, screen printing, air knife coating and so on. The thickness of the coating film is not particularly limited, but a thickness of 1 to 50 μm is preferable, and a thickness of 5 to 30 μm is more preferable.

[0029] Step (III)

Step (III) is a step of forming a lubricating film on the surface of the member by heat curing the composition applied in step (II). Thermal curing can be performed by heating in an oven or the like. When thermosetting resin is used as a binder resin, cross-linkage is formed by heating, and a cured film is formed on the member. When heating the applied composition, the solvent in the composition can be removed in the first phase of heating, and it can be cured by crosslinking reaction in the second phase of heating. For example, the first phase of heating may be performed at 60 to 100°C for 5 to 30 minutes, and then the second phase of heating may be performed at 180 to 250°C. for 20 to 120 minutes.

Examples

[0030] The present invention is illustrated by, but is not limited to, the following examples.

[0031] The raw materials used in the examples are shown in Table 1.

[0032] The "average particle diameter" in Table 1 is the average particle diameter of each particle measured by laser diffraction scattering method.

[0033]

[Table 1]

Table 1

Component	Compound name	Property	Manufactuer
(A) Binder resin			
A-1	Polyamideimide resin	Molecular weight: about 20,000	Hitachi Chemical Company, Ltd.
A-2	Epoxy resin	Molecular weight: about 400, Epoxy equivalent: 184-194	Japan epoxy resin
A-3	Polyamideimide resin	Molecular weight: about 25,000	Hitachi Chemical Company, Ltd.
(B) Hard particles			
B-1	Metal tungsten particle	Primary particles, average particle size: 1μm	Japan New Metals Co., Ltd.
B-2	Metal tungsten particle	Contains secondary particle, average particle size: 6μm	Japan New Metals Co., Ltd.
B-3	Metal tungsten particle	Contains secondary particle, average particle size: 12μm	Japan New Metals Co., Ltd.
B-4	Metal tungsten particle	Coarse tungsten powder, average particle size: 30μm	Japan New Metals Co., Ltd
B-5	Tungsten carbide particle	Average particle size: 2.6 μm	Japan New Metals Co., Ltd
B-6	Titanium disilicide particle	Average particle size: 3.4 μm	Japan New Metals Co., Ltd
B-7	zirconium carbide particle	Average particle size: 2.3 μm	Japan New Metals Co., Ltd
B-8	zirconium nitride particle	Average particle size: 1.4 μm	Japan New Metals Co., Ltd
(C) Solid lubricant			
C-1	Graphite	Average particle size: 4-5.5μm	Graphit Kropfmühl GmbH
C-2	Molybdenum disulfide	Average particle size: 4-6μm	Climax Molybdenum Co., Ltd.
(D) Solvent			
D-1	γ-butyrolactone	-	Mitsubishi Chemical Corp.
D-2	1,3-dimethyl-2-imidazolidinone (DMI)	-	Mitsui Chemical Co., Ltd.
(E) Additives			
E-1	Copolymer of ethyl methyl siloxane and 2-phenylpropyl methyl siloxane (deformer)	Refractive index: 1.46, Dynamic viscosity: 1,400cSt	Dow Corning Toray Co.,Ltd.

[0034] Example 1

A polyamideimide resin solution with about 35 wt% of solid content was obtained by dissolved 87 parts by weight of polyamideimide resin (A-1) in γ-butyrolactone (GBL, D-1). A binder resin solution was obtained by adding 13 parts by weight of epoxy resin

(A-2) in the above mentioned polyamideimide resin solution. In this binder resin solution, 55.7 parts by weight of graphite (C-1), which is a powdery solid lubricant, and 30.4 parts by weigh of metal tungsten particles (B-1), which are hard particles, and 0.7 parts by weight of a deformer (E-1) were added, then mixed and dispersed at room temperature. The mixture was diluted with GBL (D-1) and a coating composition with a solid content concentration of 47.0 wt% used for the evaluation test was formulated. The total amount of GBL (D-1) was 210.9 parts by weight. The coating composition was applied on the surface of a SRV cylindrical disk test piece (size: φ24 x 7.9 mm, Material: 100Cr6 (equivalent to SUJ2) Optimol Instruments Prutechnik GmbH by use of a screen printing plate (Mesh Corporation) so that the film thickness after baking was 15±5 μm. The film was heated and the solvent was removed by heating in a circulating oven at 80°C for 20 minutes. Thereafter, baking was performed at 220°C for 20 minutes to obtain a test piece.

[0035] Examples 2 to 3 and Comparative Examples 1 to 7

The same procedure as in Example 1 was carried out except using the components and the amount (parts by weight) shown in Table 2 or Table 3, to obtain test pieces.

[0036] The test pieces obtained in the Examples and Comparative Examples were subjected to SRV test described later, and the results are shown in Table 2 or Table 3. As shown in Table 2 or Table 3, the test pieces according to the example of the present invention had an excellent lubricating performance (small average coefficient of dynamic friction) with less depth of wear and less width of wear of corresponding material comparing to comparative examples.

[0037]

[Table 2]

Table 2

Examples		1	2	3
Binder resin	A-1	87	87	-
	A-2	13	13	-
	A-3	-	-	100
Hard particles	B-1	30.4		50
	B-2	-	30.4	-
	B-3	-	-	-
	B-4	-	-	-
	B-5	-	-	-
	B-6	-	-	-
	B-7	-	-	-
Solid lubricant	C-1	55.7	55.7	15
	C-2	-	-	80
Solvent	D-1	210.9	210.9	-
	D-2	-	-	730
Additive	E-1	0.7	0.7	0.7
Binder content (wt %)		25.1	25.1	10.2
Solid content (wt %)		47.0	47.0	25.2
Depth of wear scar (μm)		0.9	0.4	1.6
Width of wear scar (mm)		0.32	0.43	0.31
Average coefficient of dynamic friction		0.12	0.12	0.11

[0038]

[Table 3]

Table 3

Comparative examples		1	2	3	4	5	6	7
Binder resin	A-1	87	87	87	-	-	-	-
	A-2	13	13	13	-	-	-	-
	A-3	-	-	-	100	100	100	100
Hard particles	B-1	-		-	-	-	-	-
	B-2	-	-	-	-	-	-	65.2
	B-3	-	30.4	-	-	-	-	-
	B-4	-	-	30.4	-	-	-	17.4
	B-5	-	-	-	50	-	-	-
	B-6	-	-	-	-	50	-	-
	B-7	-	-	-	-	-	50	-
Solid lubricant	C-1	55.7	55.7	55.7	-	-	-	-
	C-2	-	-	-	80	80	80	80
Solvent	D-1	210.9	210.9	210.9	-	-	-	-
	D-2	-	-	-	730	730	730	730
Additive	E-1	0.7	0.7	0.7	0.7	0.7	0.7	0.7
Binder content (wt %)		27.2	25.1	25.1	10.4	10.4	10.4	9.6
Solid content (wt %)		42.6	47.0	47.0	24.0	24.0	24.0	30.0
Depth of wear scar (μm)		2.8	5.2	8.9	0.1	2.7	5.4	4.2
Width of wear scar (mm)		0.18	0.62	0.65	0.69	0.52	0.45	0.47
Average coefficient of dynamic friction		0.10	0.15	0.16	0.15	0.14	0.14	0.15

[0039] Evaluation method

Using the SRV friction and wear tester (Optimol Instruments Prutechnik GmbH, product name: oscillation friction and wear tester Model SRV Type 5), evaluation was conducted by the method in which the SRV cylinder reciprocated on the surface of the SRV disc test piece. The cylinder, it is a corresponding material, was φ15 x 22 mm SRV cylinder (Optimol Instruments Prutechnik GmbH, material: 100Cr6 (equivalent to SUJ2)). The SRV disk on which a film made of the composition of the present invention was formed was set on the test bed of the SRV5 tester, and 0.1 g of an engine oil (Exxon Mobil, chemically synthesized oil for gasoline and diesel engines 10W-30 SM/CF) was dropped on the film. Sliding the SRV cylinder for 12 hours under pressed with a load of 20 N, at a speed of 30 Hz and a sliding distance of 2 mm. Coefficient of friction was measured for each 1 hour. After that, engine oil was removed, the depth of wear scar was measured by surface roughness measurement equipment (SURFCOM1400D) by the comparison between the heights of sliding part and un-sliding part. The SRV cylinder, which is a corresponding material, was observed using microscope to determine the width of wear scar. The average coefficient of dynamic

friction was calculated from the average of the coefficient of friction measured for each 1 hour.

Claims

[Claim 1] A composition, comprising:

(A) a binder resin;

(B) a hard particle; and

(C) a solid lubricant;

wherein the hard particle comprises metal tungsten particles with 0.5 to 10 micrometers of average particle size measured by laser diffraction scattering method.

[Claim 2] The composition according to claim 1, wherein the solid lubricant is selected from the group consisting of molybdenum disulfide or graphite.

[Claim 3] The composition according to claim 1, wherein the binder resin is selected from the group consisting of polyamideimide, polyimide, epoxy resin, phenol resin, polyamide, polybenzimidazole, polyphenyl sulfonate and polyether ether ketone.

[Claim 4] The composition according to claim 1, further comprises (D) a solvent.

[Claim 5] A film comprising (A) a binder resin; (B) a hard particle and (C) a solid lubricant, wherein the hard particle comprises metal tungsten particles with 0.5 to 10 micrometers of average particle size measured by laser diffraction scattering method.

[Claim 6] The film according to claim 5, wherein the solid lubricant is selected from the group consisting of molybdenum disulfide or graphite.

[Claim 7] The film according to claim 5, wherein the binder resin is selected from the group consisting of polyamideimide, polyimide, epoxy resin, phenol resin, polyamide, polybenzimidazole, polyphenyl sulfonate and polyether ether ketone.

[Claim 8] A sliding member having the film any of claims 5 to 7.

[Claim 9] The sliding member according to claim 8, wherein the sliding member is a swash plate of a compressor, an engine tappet, a camshaft, a crankshaft, an engine metal, an engine piston, a piston ring, a gear, a door lock, a brake shim or a brake clip.

[Claim 10] A method for forming a lubricating film on surface of a member, comprising the steps of:

(I) preparing a composition comprising (A) a binder resin, (B) a hard particle, and (C) a solid lubricant, wherein the hard particle comprises metal tungsten particles with 0.5 to 10 micrometers of average particle size measured by laser diffraction scattering method,

(II) applying the composition on surface of a member; and

(III) heat-curing the applied composition, thereby forming a lubricating film on the surface of the member.

INTERNATIONAL SEARCH REPORT

International application No
PCT/JP2019/047025

A. CLASSIFICATION OF SUBJECT MATTER
INV. C08K3/04 C08K3/08 C08K3/30 F16C33/00 B32B27/08
C08J5/18
ADD.
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)
C08K F16C C08J B32B

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

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, CHEM ABS Data, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 3 346 031 A1 (GARWARE WALL ROPES LTD [IN]) 11 July 2018 (2018-07-11) abstract; claims 1,9-11; example 5	1,2
A	EP 2 530 317 A1 (PERKINS ENGINES CO LTD [GB]) 5 December 2012 (2012-12-05) abstract; claims 1,9,10	1-10
A	EP 2 048 392 A2 (DAIDO METAL CO [JP]) 15 April 2009 (2009-04-15) abstract; claims 1-3 paragraph [0018]	1-10
A	EP 1 894 987 A1 (HONDA MOTOR CO LTD [JP]) 5 March 2008 (2008-03-05) abstract; claim 1	1-10

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
19 February 2020	28/02/2020

Name and mailing address of the ISA/	Authorized officer
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/JP2019/047025

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