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(54) **METHOD FOR COATING A METAL PART DESTINED TO BE SUBJECTED TO HIGH CONTACT PRESSURES AND METAL PART OBTAINED THEREFROM**

(57) The invention relates to a method for coating a metal part (P) destined to be subjected to high contact pressures, an external layer of the metal part (P) forming a substrate (S) to be coated, which comprises the steps consisting in a) Applying a nickel strike treatment to the substrate (S), such that a first layer (1) of nickel is obtained; b) Applying a copper electrolytic plating to the first layer (1) of nickel, such that a second layer (2) of copper is obtained; c) Applying a tin plating to the second layer (2) of copper, such that a third layer (3) is obtained, the interface between the second layer (2) and the third layer (3) being bronze. The invention also relates to coated parts using said method, the coated parts being preferably a pin and/or a box of a threaded joint for oil and gas applications

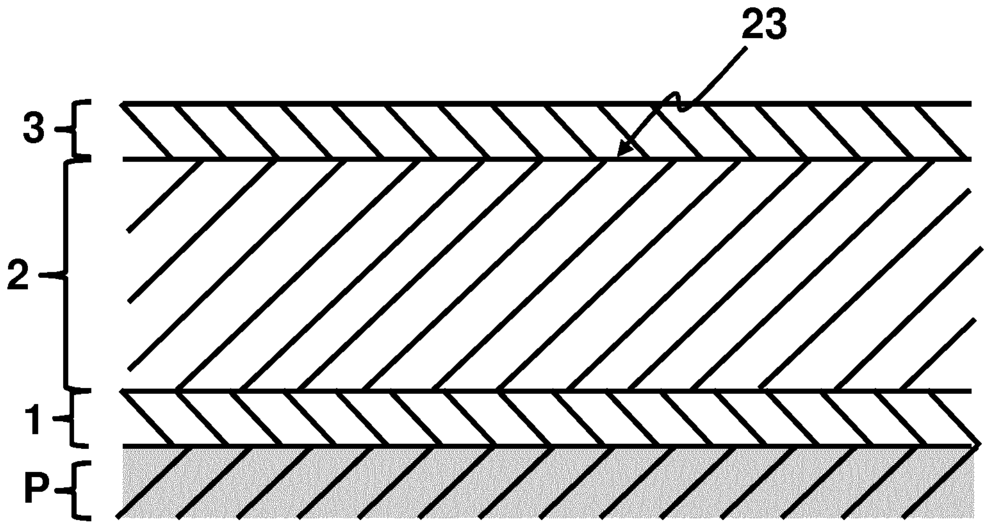

Fig. 1

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Description

TECHNICAL FIELD

[0001] The present invention relates to coatings. In particular, to coatings that improve anti-galling and frictional properties of metal parts.

STATE OF THE ART

[0002] Threaded joints of oil and gas ducts are subjected to high contact pressures that cause the opposite contact surfaces to bond, i.e. to weld in the atomic level. Since the joined parts have to move relative to each other during make and breaks, this bond may lead to galling of the contact surfaces.

[0003] The production of galling can be minimized by reducing the stiction and coefficient of friction between the parts.

[0004] A solution to prevent galling is to coat the parts. Among the coating methods, electrolytic plating and electroless plating are known.

[0005] The electrolytic deposition techniques are based on the application of electric current. One of the advantages of these known techniques is the speed of deposition, which makes them particularly suitable when it comes to depositing relatively thick layers, such as, for example, of the order of 10 μm.

[0006] In contrast, in the electroless plating the coating is obtained without the application of current. This technique allows for obtaining very uniform coatings, especially when the coating to be deposited is relatively thin, say 1 μm. A major advantage of the uniformity is that it results in an improved performance of the joint.

[0007] An example of electroless coating is disclosed in US 4,527,815 (Frick). This document discloses an electroless nickel coating, consisting, immersing the part to be coated in a hot bath containing an organic nickel compound.

[0008] US 4,758,025 (Frick) also discloses the use of electroless metal coatings, specifically copper or zinc coatings, but again no details are provided regarding the specific composition of the bath and the full method.

[0009] US2015159277 discloses an aqueous electroless nickel plating bath for forming electroless nickel coatings that includes nickel, a hypo phosphorous reducing agent, zinc, a bismuth stabilizer, and a complexing agent.

[0010] US 2004/0086656 (Kohl et al) relates to electroless copper plating and methods of use thereof, in fields away from oil and gas pipes like PCB manufacture, and mentions the use of an accelerator for accelerating copper deposition. Growth rates are a major parameter to be optimized, since it is very relevant for the manufacturing costs.

[0011] WO8201015A1 (Shipley company Inc.) discloses combinations of copper/ nickel electroless coating where copper amounts to up to 70%, nickel being excluded from being an important part of the coating due to its bad electrical conducting properties.

[0012] In any case, what is sought is to decrease the galling by reducing the coefficient of friction.

[0013] A secondary objective is to reduce the use of lubricating greases containing polluting metals.

DESCRIPTION OF THE INVENTION

[0014] For overcoming the low yields and long times for obtaining an anti-galling coating, the invention provides an efficient method for coating a metal part destined to be subjected to high contact pressures, an external surface of the metal part forming a substrate to be coated, which comprises the following steps:

a) Applying a nickel strike treatment to the substrate, such that a first layer of nickel is obtained;
b) Applying a copper electrolytic plating to the first layer of nickel, such that a second layer of copper is obtained;
c) Applying a tin plating to the second layer of copper, such that a third layer is obtained, the interface between the second layer and the third layer being bronze.

[0015] The proposed method allows for reducing the stiction and coefficient of friction, and thus improving the anti-galling properties and corrosion resistance. Moreover, thanks to the stiction and coefficient of friction reduction, it is possible to use non-contaminant greases while keeping the performance of lead containing greases. In particular, the friction or anti-galling properties of the coating produced satisfies the standard requirements when an environmentally friendly lubricant is used.

[0016] The proposed method achieves its beneficial effects thanks to the combination of three clearly differentiated layers, as well as the created bronze interface.

[0017] The electrolytic plating has proved in the present case to be an efficient process to generate the thickness required for the second layer.

[0018] The method is especially efficient when is applied to carbon steel, stainless steel or nickel-based alloys, which are generally used for OCTG (Oil Country Tubular Goods) pipes in API5CT, API5CRA and API6A standards.

[0019] In an embodiment, the step a) of the method is carried out with an immersion in a solution containing hydrochloric acid (HCl) and nickel chloride hexahydrate ($NiCl_2 \cdot 6H_2O$).

[0020] This is a common way to Nickel coating that has proven to combine in an efficient way with the present invention.

[0021] In a more specific embodiment, the hydrochloric acid concentration is in the range of 5% - 10% in weight and the nickel chloride hexahydrate is in the range of 150 - 300 g/L, the anode being nickel having at least a 99.5% purity.

[0022] This concentration allows to improve significantly the adhesion of the subsequent layer.

[0023] In an embodiment, the thickness of the first layer is comprised in the range of 0.1-1 μm. This thickness provides a good bonding between coating film and substrate material.

[0024] The nickel strike is processed at room temperature. The thickness is controlled by the following equation,

$$Thickness = \eta \frac{It}{2F\rho S} \quad (1)$$

where η is the nickel strike efficiency, n=40% (this value is various to the concentration of HCl: the higher HCl, the lower η); I is the electrical current; t is plating time; F is the Faraday constant, F=96485 C/mol; ρ is the density of nickel; S is the surface area of the substrate.

[0025] In an embodiment, the electrolytic plating is carried out in a solution containing copper sulphate pentahydrate ($CuSO_4 \cdot 5H_2O$) and sulfuric acid (H_2SO_4).

[0026] In a more specific embodiment:

- the $CuSO_4 \cdot 5H_2O$ has a concentration comprised in the range of 100 - 200 g/L;
- the H_2SO_4 has a concentration comprised in the range of 20 - 100 g/L; and
- the anode being an oxygen free phosphorous copper (OFC), with no less than 99.5% pure copper metal. The phosphorous content is in range of 0.01%-0.08%.

[0027] An advantage is that the coating does not delaminate while allowing a high yield coating method.

[0028] Another advantage is that it allows to control the thickness of the deposited copper layer, ensuring an appropriate adhesion strength.

[0029] In an embodiment, the thickness of the second layer is comprised in the range of 6 - 12 μm, and is preferably 10 μm.

[0030] In an embodiment, the step c) comprises an electroless coating.

[0031] The advantage of electroless method is to deposit a uniform layer of coating, regardless of the surface profile of substrate and avoid the 'shielding' effect in electrolytic plating process.

[0032] A further advantage of the electroless method is that it allows to control the thickness of the coating of a rough surface.

[0033] In an embodiment, the electroless coating is applied with a solution containing stannous sulphate ($SnSO_4$), thiourea (CH_4N_2S) and sulfuric acid (H_2SO_4).

[0034] In a more specific embodiment:

- the $SnSO_4$ has a concentration comprised in the range of 1 - 10 g/L;
- the thiourea has a concentration comprised in the range of 20 - 100 g/L; and
- the H_2SO_4 has a concentration comprised in the range of 10 - 50 g/L.

[0035] In an embodiment, the thickness of the third layer is comprised in the range of 0,4 -- 0,6 μm and is preferably 0,5 μm.

[0036] In an embodiment, the method comprises a further step d) consisting in baking the obtained coating at 150-200 °C.

[0037] The baking at 150-200 °C improves the coating quality by two reasons: a) releasing residual stress; b) strengthening the third layer itself and the bonding with the copper layer underneath.

[0038] In an embodiment, the metal part is a pin and/or a box of a threaded joint for oil and gas applications.

[0039] In another aspect, the present invention provides a metal part destined to be subjected to high contact pressures, the metal part having an external layer which forms a substrate, the substrate having a coating, wherein the coating comprises:

- a first layer of nickel on the substrate;
- a second layer of copper on the first layer;
- a third layer of tin on the second layer;

- the interface between the second layer and the third layer being bronze;

wherein:

- the thickness of the first layer is comprised in the range of 0.1-1 μm;
- the thickness of the second layer is comprised in the range of 6 - 12 μm, and is preferably 10 μm; and
- wherein the thickness of the third layer is comprised in the range of 0,4 and 0,6 μm and is preferably 0,5 μm.

[0040] Preferably, the metal part is a pin and/or a box of a threaded joint for oil and gas applications

BRIEF DESCRIPTION OF THE DRAWINGS

[0041] To complete the description and in order to provide for a better understanding of the invention, a set of drawings is provided. Said drawings form an integral part of the description and illustrate embodiments of the invention, which should not be interpreted as restricting the scope of the invention, but just as an example of how the invention can be carried out. The drawings comprise the following figures:

Figure 1 schematically shows the coating obtained when applying the method according to the present invention.

Figure 2 is a graph showing the evolution of the coefficient of friction as a function of the compression cycles, which has been obtained in a test bench and using API as a lubricating grease in a pin box contact.

Figure 3 is a graph showing the evolution of the coefficient of friction as a function of the compression cycles, which has been obtained in a test bench and using green dope as a lubricating grease in a pin box contact.

DESCRIPTION OF A WAY OF CARRYING OUT THE INVENTION

[0042] The present invention relates to a method for coating a metal part P destined to be subjected to high contact pressures, in particular to pin and/or a box of a threaded joint for oil and gas applications.
[0043] The metal part P forms the substrate S to be coated.
[0044] Specifically, and with reference to FIG. 1, the method comprises the following steps:

a) Applying a nickel strike treatment to the substrate S, such that a first layer 1 of nickel is obtained;
b) Applying a copper electrolytic plating to the first layer 1 of nickel, such that a second layer 2 of copper is obtained;
c) Applying a tin plating to the second layer 2 of copper, such that a third layer 3 is obtained, the interface 23 between the second layer 2 and the third layer 3 being bronze.

[0045] Step a is carried out with a bath in a solution containing hydrochloric acid HCl and nickel chloride hexahydrate $NiCl_2 \cdot 6H_2O$, wherein the hydrochloric acid concentration is in range 5% - 10% in weight and the nickel chloride hexahydrate is in the in range 150 - 300 g/L, the anode being nickel having at least a 99.5% purity.
[0046] The first layer 1 is comprised in the range of 0.1-1 μm.
[0047] The electrolytic plating is carried out in a solution containing copper sulphate pentahydrate $CuSO_4 \cdot 5H_2O$ and sulfuric acid H_2SO_4, wherein:

- the $C_uSO_4 \cdot 5H_2O$ has a concentration comprised in the range of 100 - 200 g/L;
- the H_2SO_4 has a concentration comprised in the range of 20 - 100 g/L; and
- the anode being an oxygen free phosphorous copper OFC, with no less than 99.5% pure copper metal. The phosphorous content is in range of 0.01%-0.08%.

[0048] The electronic current density on anode should be less than 5 A/cm^2 without agitation, or 17 A/cm^2 with agitation.

Name	Formula		
Copper sulphate pentahydrate	$CuSO_4 \cdot 5H_2O$	200	g/L
Sulfuric acid	H_2SO_4	50	g/L

[0049] The coating thickness is calculated by the equation 1, however, the plating efficiency η=1.

[0050] The second layer 2 is comprised in the range of 6 - 12 μm, and is preferably 10 μm.

[0051] Step c is an electroless coating applied with a solution containing stannous sulphate $SnSO_4$, thiourea CH_4N_2S and sulfuric acid H_2SO_4, and wherein:

- the $SnSO_4$ has a concentration comprised in the range of 1 - 10 g/L;
- the thiourea has a concentration comprised in the range of 20 - 100 g/L; and
- the H_2SO_4 has a concentration comprised in the range of 10 - 50 g/L.

[0052] Step c) has to be applied such that the thickness of the third layer 3 is comprised in the range of 0,4 and 0,6 μm and is preferably 0,5 μm.

[0053] The electroless process applies a very thin layer of tin on top of the copper layer. The electroless bronze solution contains stannous sulphate (S_nSO_4), thiourea (CH_4N_2S) and sulfuric acid (H_2SO_4). The thiourea reduces the electrode potential of copper, so that it reduces stannous ion, and then an ultrathin tin layer is formed on top of copper.

Name	**Formula**	**g/L**	**mol/L**
Stannous sulphate	S_nSO_4	4.5	0.021
Thiourea	CH_4N_2S	50	0.658
Sulfuric acid	H_2SO_4	20	0.204

[0054] The S_nSO_4 and CH_4N_2S must be dissolved in H_2SO_4 and deionized water respectively first, and then mixed together.

[0055] The electroless bronze can be processed at room temperature. The deposition initiates automatically when the part to be coated is immersed into the electroless bronze solution. After a few minutes (about 2 minutes), the process is stopped automatically when all the surface of copper is covered by tin.

[0056] Although the coated parts P obtained can be used in dry-type joints, their preferred use will be with in wet joints, that is with the use of a lubricating grease interposed between the two parts (threaded pin and box for oil and gas applications) subjected to high mutual pressures.

[0057] In the present case, two types of greased, API and green dope, where tested.

[0058] These two differ in their effect on the environment. While API is harmful due to its heavy metal content, the green dope is respectful of the environment.

[0059] The use of the former, API, in a metal-to-metal friction results, in combination with known coatings, in a reduced coefficient of friction.

[0060] When the coating according to the present invention is used, a considerable reduction in the coefficient of friction is achieved, which becomes more evident as the cycles of use accumulate. We emphasize that the pin-box joints are subjected to high pressures during make and breaks that cause a relative movement between the parts P.

[0061] However, it has been found that the reduction in the coefficient of friction also occurs when using greases that do not contain heavy metals such as the green dope, reaching performances that make their performance comparable in a pin-box joint with known coatings in combination with the use of API, which is a contaminant to the environment.

[0062] Figure 2 represents the evolution of the coefficient of friction after applying several pressure cycles with the tests disclosed in "Characterisation of friction and lubrication regimes in premium tubular connections" from F. Stewart, H.R.Le, J.A.Williams, A.Leech, B.Bezensek, A.Roberts.

[0063] The objective of this test is to determine the behavior of the coating in the long term and what is obtained is the surprising result illustrated.

[0064] Specifically, it is observed that in the first cycles the behavior as far as friction is concerned is comparable.

[0065] However, it is appreciated that the difference is widened as cycles accumulate, with the order reduction being approximately 15%.

[0066] When comparing the results obtained with different greases, it is appreciated that it does not depend on these. FIG. 3 is the graph obtained with the use of green dope. The result is maintained in a reduction with the aforementioned percentage, i.e. 15%.

[0067] In this text, the term "comprises" and its derivations (such as "comprising", etc.) should not be understood in an excluding sense, that is, these terms should not be interpreted as excluding the possibility that what is described and defined may include further elements.

[0068] The invention is obviously not limited to the specific embodiments described herein, but also encompasses any variations that may be considered by any person skilled in the art within the general scope of the invention as defined in the claims.

Claims

1. Method for coating a metal part (P) destined to be subjected to high contact pressures, an external layer of the metal part (P) forming a substrate (S) to be coated, which comprises the following steps:

   a) Applying a nickel strike treatment to the substrate (S), such that a first layer (1) of nickel is obtained;
   b) Applying a copper electrolytic plating to the first layer (1) of nickel, such that a second layer (2) of copper is obtained;
   c) Applying a tin plating to the second layer (2) of copper, such that a third layer (3) is obtained, the interface between the second layer (2) and the third layer (3) being bronze.

2. Method according to claim 1, wherein step a) is carried out in a bath with a solution containing hydrochloric acid (HCl) and nickel chloride hexahydrate ($NiCl_2 \cdot 6H_2O$).

3. Method according to claim 2, wherein the hydrochloric acid concentration is in the range of 5% -- 10% in weight and the nickel chloride hexahydrate is in the range of 150 - 300 g/L, the anode being nickel having at least a 99.5% purity.

4. Method according to any of the preceding claims, wherein the thickness of the first layer (1) is comprised in the range of 0.1 -- 1 μm.

5. Method according to any of the previous claims, wherein the electrolytic plating is carried out in a solution containing copper sulphate pentahydrate ($CuSO_4 \cdot 5H_2O$) and sulfuric acid (H_2SO_4).

6. Method according to claim 5, wherein:

   - the $CuSO_4 \cdot 5H_2O$ has a concentration comprised in the range of 100 - 200 g/L;
   - the H_2SO_4 has a concentration comprised in the range of 20 - 100 g/L; and
   - the anode being an oxygen free phosphorous copper (OFC), with no less than 99.5% pure copper metal, the phosphorous content thereof being in the range of 0.01%-0.08%.

7. Method according to any of the preceding claims, wherein the thickness of the second layer (2) is comprised in the range of 6 - 24 μm, and is preferably 10 μm.

8. Method according to any of the previous claims, wherein step c) is an electroless coating.

9. Method according to claim 8, wherein the electroless coating is applied with a solution containing stannous sulphate ($SnSO_4$), thiourea (CH_4N_2S) and sulfuric acid (H_2SO_4).

10. Method according to claim 9, wherein:

    - the $SnSO_4$ has a concentration comprised in the range of 1 - 10 g/L;
    - the thiourea has a concentration comprised in the range of 20 - 100 g/L; and
    - the H_2SO_4 has a concentration comprised in the range of 10 - 50 g/L.

11. Method according to any of the previous claims, wherein the thickness of the third layer (3) is comprised in the range of 0,2 to 0,8 μm and is preferably 0,5 μm.

12. Method according to any of the previous claims, which comprises a further step d) consisting in baking the obtained coating at 150 -- 200 °C.

13. Method according to any of the previous claims wherein the metal part (P) is a pin and/or a box of a threaded joint for oil and gas applications.

14. Metal part (P) destined to be subjected to high contact pressures, the metal part (P) having an external layer which forms a substrate (S), the substrate (S) having a coating, **characterized in that** the coating comprises:

    • a first layer (1) of nickel on the substrate (S);
    • a second layer (2) of copper on the first layer (1);

- a third layer (3) of tin on the second layer (2);
- the interface between the second layer (2) and the third layer (3) being bronze;

wherein:

- the thickness of the first layer (1) is comprised in the range of 0.1--1 μm;
- the thickness of the second layer (2) is comprised in the range of 6 - 24 μm, and is preferably 10 μm; and
- wherein the thickness of the third layer (3) is comprised in the range of 0,2 --0,8 μm and is preferably 0,5 μm.

15. Metal part (P) according to claim 14, the metal part being a pin and/or a box of a threaded joint for oil and gas applications.

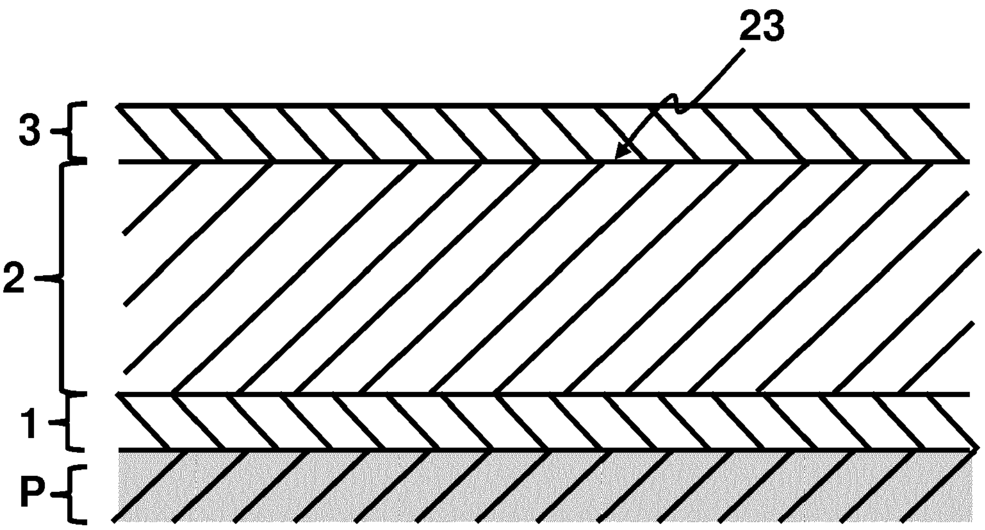
23
3
2
1
P

Fig. 1

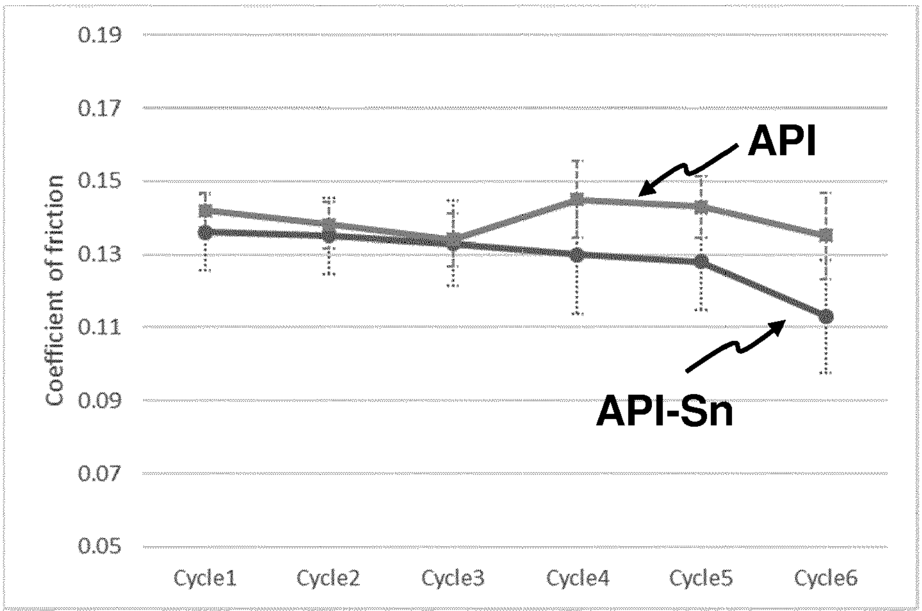
0.19
0.17
0.15
0.13
0.11
0.09
0.07
0.05
Coefficient of friction
API
API-Sn
Cycle1
Cycle2
Cycle3
Cycle4
Cycle5
Cycle6

Fig. 2

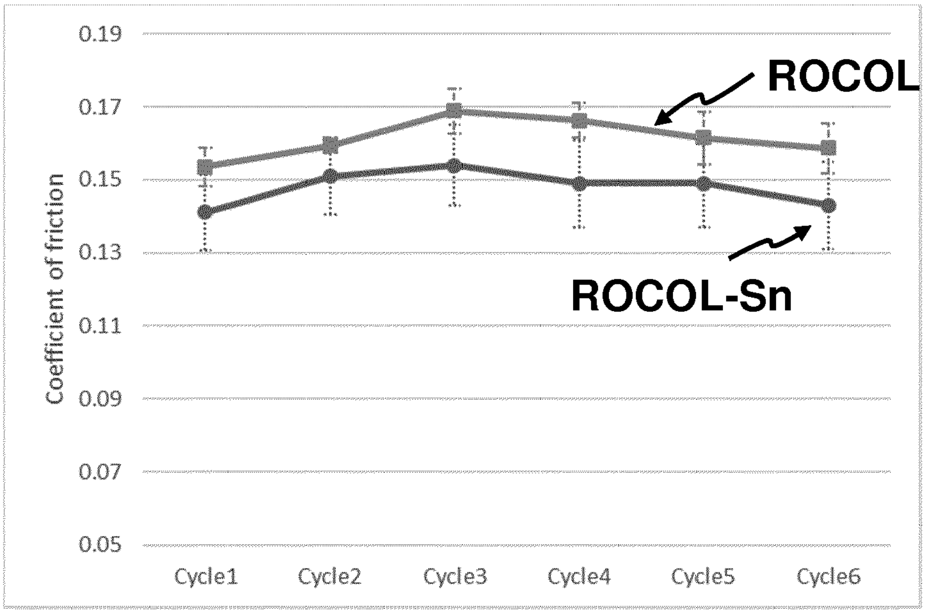
0.19
0.17
0.15
0.13
0.11
0.09
0.07
0.05
Coefficient of friction
Cycle1
Cycle2
Cycle3
Cycle4
Cycle5
Cycle6
ROCOL
ROCOL-Sn

Fig. 3

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EUROPEAN SEARCH REPORT

Application Number
EP 18 38 2639

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The present search report has been drawn up for all claims

2

Place of search	Date of completion of the search	Examiner
The Hague	8 February 2019	Picard, Sybille

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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EPO FORM 1503 03.82 (P04C01)

ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO. EP 18 38 2639

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

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