

Specification

Method for surface treatment of a steel mechanical part to improve its resistance to wear and corrosion under harsh stresses

Technical Field

5 [0001] The present invention relates to the field of surface treatment of steel mechanical parts intended to improve their resistance to wear and corrosion. More particularly, the invention concerns a treatment process involving nitriding or nitrocarburizing, optionally followed by oxidation, and impregnation with a hydrophobic wax.

10 Prior Art

[0002] Document EP0524037 discloses a surface treatment method for improving the corrosion resistance of mechanical parts.

[0003] This document describes a technique for enhancing the friction and corrosion resistance properties of ferrous metal parts. The disclosed technique involves a series of treatments, including nitriding, oxidation, and the application of a final coating.

15 [0004] This final coating is a hydrophobic wax, an organic carbon-based compound having a high molecular weight, notably between 500 and 10,000. The application of this high-molecular-weight wax helps improve the corrosion resistance of the parts and minimize rebound effects.

[0005] These treatments are advantageous in that they are inexpensive and easy to implement, even for industrial-scale production of series parts. In addition, they provide the treated parts with high technical performance even for complex shapes.

25 [0006] Document WO2016/102813 discloses a process using PVD coating to increase the durability of mechanical parts.

[0007] This document describes a method for treating the surface of a steel mechanical part to provide it with high resistance to wear and corrosion. The process comprises the following steps:

- a nitriding or nitrocarburizing step to form an iron nitride layer at least 8 micrometers thick;

- an oxidation step to generate an oxide layer between 0.1 and 3 micrometers thick;
- an impregnation step by immersion in an impregnation bath for at least 5 minutes. The bath consists of at least 70% by weight, +/-1%, of a hydrocarbon solvent, 10% to 30% by weight, +/-1%, of at least one paraffin oil, and at least one synthetic phenolic additive at a concentration between 0.01% and 3% by weight, +/-0.1%.

[0008] This method achieves a substantial improvement in corrosion resistance compared to a conventional oil- or acid-based bath. Furthermore, the treated parts are dry to the touch, meaning they do not transfer oil to another surface or attract dust. This also allows for post-treatments such as overmolding.

[0009] However, with either of the aforementioned techniques, if the treated and impregnated part is subjected to severe conditions such as high-pressure washing or repeated mechanical stresses (e.g., friction by a seal), corrosion resistance is degraded.

[0010] To address this issue, it is known to use specific coating technologies such as electroless nickel plating or hard chrome coating. However, these techniques have drawbacks, including:

- Fluctuation in raw material costs: Nickel, for instance, is subject to significant price variations due to economic and geopolitical factors, making the cost of coating operations unpredictable and potentially more expensive;
- REACH regulation: The European Regulation on the Registration, Evaluation, Authorization and Restriction of Chemicals (REACH) subjects the use of certain materials, such as hard chrome, to prior authorization. This can slow production and increase costs. There are also environmental and health concerns associated with these substances.

[0011] Consequently, although these coating technologies can improve the wear and corrosion resistance of steel mechanical parts, they face challenges in terms of cost and regulatory compliance.

[0012] Document FR3030578 also describes the current state of the art.

Summary of the Invention

[0013] One objective of the invention is to overcome the disadvantages of the prior art by providing a surface treatment process for a steel mechanical part that improves its resistance to wear and corrosion, maintaining this resistance even when subjected to aggressive and severe environmental conditions such as repeated high-pressure washing, exposure to sand and weather, or repeated mechanical friction.

[0014] The present invention solves the problems of the prior art by proposing a surface treatment process for a steel mechanical part comprising at least one nitriding or nitrocarburizing step forming a nitrided surface layer, for example comprising a dense inner sublayer and a porous outer sublayer. The outer sublayer contains more than 60% Fe_{2-3}N solid phase and has a hardness between 550 and 650 HV 0.1. Its roughness is between 0.3 and 1.5 micrometers, which further improves wax adhesion and corrosion resistance. This nitriding or nitrocarburizing step is followed by an impregnation step of this layer with a solution comprising at least one hydrophobic wax.

[0015] According to the invention, the wax is an organic carbon-based compound having a low molecular weight, specifically below 500, and preferably between 200 and 500.

[0016] This feature provides effective corrosion protection while remaining economically viable.

[0017] Moreover, contrary to the teaching of the prior art, which encouraged the use of high-molecular-weight organic carbon compounds, the Applicant has discovered that, provided nitriding or nitrocarburizing is properly performed, a low-molecular-weight organic carbon compound enhances the retention of the protective wax and reduces its susceptibility to washing or degradation under severe use conditions.

[0018] Under these conditions, the invention ensures superior corrosion resistance and high resistance to high-pressure washing/cleaning, exposure to sand and weather, or repeated mechanical friction.

[0019] The invention considers satisfactory corrosion resistance to be greater than 1600 hours in a salt spray test for a freshly treated part, and greater than 500 hours for a part subjected to simulated high-pressure washing after treatment.

[0020] Impregnation according to the invention does not alter the visual appearance or handling of the parts and provides a dry-to-touch surface.

[0021] The process may further include an oxidation step following nitriding or nitrocarburizing, resulting in the formation of a porous surface layer containing iron oxides. This oxide layer enhances corrosion resistance and wax retention.

5 [0022] The impregnation can be carried out by immersion in a bath or spraying the solution at room temperature, making the process flexible and adaptable to different part geometries.

[0023] The immersion time is at least 2 minutes, which provides an effective compromise between treatment time and protection quality.

10 [0024] Given the observed performance, the impregnation solution can contain wax diluted with solvent to 50%, 75%, or 80%, still achieving exceptional corrosion resistance under severe conditions. When diluted to 90%, the wax remains protective under normal conditions but no longer suitable for severe conditions.

15 [0025] The process advantageously requires no oven drying, air drying the impregnated solution for at least 5 minutes at room temperature is sufficient and ensures good wax adhesion.

[0026] The solution advantageously contains calcium sulfonate in an amount between 1% and 30% by mass, preferably between 1% and 20%, improving corrosion resistance while being environmentally friendly.

20 [0027] Calcium sulfonate also acts as a lubricant, reducing friction and wear between moving surfaces. When added to the wax, it improves the wax's lubricating properties and promotes smoother motion, reducing, for example, seal scraping on a hydraulic rod.

25 [0028] The addition of calcium sulfonate also enhances the wax's oxidation resistance, acting as an antioxidant that prevents wax degradation in the presence of oxygen, thus extending wax life.

[0029] Calcium sulfonate further provides water stability, forming a protective layer on metal surfaces to prevent corrosion and maintain lubrication even in humid environments.

30 [0030] In one embodiment, the solution comprises a wax essentially consisting of a C20–C35 alkane fraction and calcium sulfonate.

[0031] This provides excellent corrosion protection while using economical and readily available raw materials.

[0032] The wax can be diluted with a solvent containing mainly C8–C12 alkanes (notably C9, C10, and C11) or with white spirit.

- 5 [0033] The invention also relates to a steel mechanical part having undergone such surface treatment, comprising a nitrided surface layer impregnated with a hydrophobic wax-based solution, where the wax is an organic carbon compound with molecular weight below 500, preferably between 200 and 500.

- 10 [0034] Preferably, based on observed performance, the treated and impregnated mechanical part can have a low wax coating weight, with a mass per unit area between 5 and 60 g/m², preferably between 30 and 60 g/m², resulting in lower product consumption, reduced cost, and limited environmental impact.

Detailed Description of the Invention

- 15 [0035] The present invention concerns a surface treatment process for steel mechanical parts to improve their resistance to wear and corrosion. This process relies on a surface treatment based on nitriding or nitrocarburizing followed by impregnation with a solution comprising at least one specific hydrophobic wax.

- 20 [0036] The invention is particularly advantageous for treating parts requiring high corrosion resistance, notably evaluated by the salt spray test (ISO 9227), and used under severe conditions such as exposure to weather, sand, or repeated high-pressure washing.

[0037] An example of a treated part is a tailgate cylinder rod located at the rear of a truck body, often exposed (extended) to environmental stress and friction from the seal at the cylinder entrance.

- 25 [0038] The nitriding or nitrocarburizing step allows diffusion of nitrogen (or nitrogen and carbon) into the steel surface, forming a nitrided layer that improves wear resistance.

[0039] In some embodiments, this step is followed by oxidation to further increase corrosion resistance by forming a protective oxide barrier.

- 30 [0040] The nitriding followed by oxidation forms a layer with a dense inner sublayer and a porous outer sublayer (5–25 µm thick) with open pores of 0.2–3 µm. The steel

parts thus treated are then impregnated with a hydrophobic wax-based solution having a molecular weight of 200–500 and a solid-state surface energy between 29 and 35 mN/m.

5 [0041] Surface energy is measured by contact angle on three polished samples immersed in wax solutions (50%, 75%, 87.5%) and dried for 24 h. The energy is nearly independent of concentration.

[0042] The wax according to the invention provides superior impregnation and resistance under severe exposure. Impregnation may be by immersion or spraying at ambient temperature.

10 [0043] Immersion lasts at least 2 minutes. The wax is generally diluted (50–80%) with solvent (C8–C12 alkanes or white spirit). After impregnation, air drying for ≥ 5 minutes completes the process.

15 [0044] The wax solution advantageously includes 1–30% calcium sulfonate, preferably 2–20%, and the wax is based on a C20–C35 alkane fraction. Optionally, a plasticizer such as 1,1'-Biphenyl 4,4'-dibromo ($C_{12}H_8Br_2$) may be added to increase cohesion.

[0045] The resulting steel part presents a nitrided layer impregnated with hydrophobic wax, significantly improving wear and corrosion resistance under severe conditions.

20 [0046] The outer sublayer contains more than 60% $Fe_{2-3}N$ phase, hardness between 550–650 HV0.1, and surface roughness 0.3–1.5 μm CLA.

[0047] The wax may be natural or synthetic (polyethylene, polypropylene, polyester, fluorinated, or modified petroleum wax).

25 [0048] The composition, thickness, and hardness of the nitrided layer are optimized to resist wear without becoming brittle or prone to flaking. The hexagonal compact structure of $Fe_{2-3}N$ offers good deformability and excellent friction performance.

30 [0049] The use of a molten salt bath, such as that described in document FR-A-2 171 993, to carry out the nitriding treatment, is a particular advantage of the present invention. This bath is essentially composed of carbonates and cyanates of the alkali metals K, Na and Li. The weight proportions of the alkali cations are 25–42.6% for

Na⁺, 42.6–62.5% for K⁺, and 11.3–17.1% for Li⁺ relative to the total weight of the bath.

[0050] The oxidation treatment also improves corrosion resistance and optimizes the surface structure for efficient wax impregnation.

- 5 [0051] Oxidation is carried out in a molten salt bath as in FR-A-2 525 637, typically at 350–450°C.

[0052] Corrosion resistance tests were performed on parts treated according to the invention and comparative parts, both washed and unwashed.

[0053] Nitriding and oxidation steps were identical; only the wax type varied.

- 10 [0054] The nitrided layer (compact + porous sublayers) was 20–30 µm thick (5–10 µm porous surface). The oxide layer was ~1–2 µm thick. All impregnations were by immersion.

[0055] High-pressure washing was performed with a 180-bar cold water jet at 50 cm distance for 30 seconds.

- 15 [0056] The results of these tests are compiled in the table below:

[0057] [Table 1]

Test	Wax	Molecular Weight	Weight Before (g)	Weight After (g)	Coating Weight (g)	Coating Weight (g/m ²) ²	Corrosion Resistance (after high-pressure washing) (NSST ISO 9227)	Corrosion Resistance (without high-pressure washing) (NSST ISO 9227) (NSST ISO9227)
0	According to the invention, diluted to 90% with white spirit	200-500	88,73	89,054	0,324	9,5	200	> 1608 hours
0 bis	According to the invention, diluted to 75% with white spirit	200-500	88,91	89,704	0,794	23,2	> 1608 hours	> 1608 hours

0 ter	According to the invention, diluted to 50% with a C8–C12 alkane fraction	200-500	86,84	87,98	1,14	33,3	> 1608 hours	> 1608 hours
1	Wax from Example 2 of patent EP0524037, diluted to 90% with white spirit	500-10.000	86,22	87,0725	0,8525	25	200 hours	700 hours
2	Wax from Example 2 of patent EP0524037, diluted to 50%	500-10.000	86,36	89,668	3,308	97	300 hours	> 1608 hours
3	Polyethylene wax, diluted to 90% with white spirit	3000-10.000	86,18	86,998	0,818	24	144 hours	700 hours
4	Polyethylene wax, diluted to 50% with white spirit	3000-10.000	86,44	90,089	3,649	107	300 hours	> 1608 hours

[0058] For each test, trials were carried out on 10 parts, and the time to the onset of corrosion on 50% of the parts was measured.

[0059] It is observed that parts 0, 0bis and 0ter, treated according to the invention and not washed, exhibit improved corrosion resistance, achieving exposure in salt spray for more than about 1600 hours without any manifestation of corrosion.

[0060] The parts 0, 0bis and 0ter, treated according to the invention and subjected to high-pressure washing, withstand salt-spray exposure in excess of 500 hours, and even in excess of about 1600 hours, without any manifestation of corrosion, which is very satisfactory, even though the amount of wax present on the layer is relatively low, namely below 60 g/m², and even below 35 g/m².

[0061] Conversely, it is noted that the corrosion resistance of parts treated with a wax in the form of an organic carbon-based compound having a molecular weight between 500 and 10,000 (see tests 1, 2, 3 and 4) is less than 500 hours after high-pressure washing and is therefore not considered satisfactory within the meaning of the present invention.

[0062] It is also observed that increasing the coating weight of the wax in Examples 2 and 4 somewhat improves corrosion resistance after high-pressure washing, but does not reach a satisfactory level of resistance.

5 [0063] From the foregoing it follows that the invention indeed provides a surface treatment process for a steel mechanical part to improve its resistance to wear and corrosion under severe service conditions, as well as such a part obtained by the process.

Claims

1. A method for surface treatment of a steel mechanical part to improve its resistance to wear and corrosion, comprising at least one nitriding or nitrocarburizing step forming a nitrided surface layer, followed by impregnation of said nitrided layer with a solution comprising at least one hydrophobic wax, characterized in that the wax is an organic carbon-based compound having a molecular weight below 500, and preferably between 200 and 500.
2. Method of claim 1, wherein the nitriding or nitrocarburizing step is followed by oxidation.
3. Method of any preceding claim, wherein impregnation is performed by immersion or spraying at ambient temperature.
4. Method of claim 3, wherein impregnation by immersion lasts at least 2 minutes.
5. Method of any preceding claim, wherein the impregnation solution comprises wax diluted with solvent to 50%, 75%, or 80%.
6. Method of any preceding claim, wherein the impregnated part is air-dried for at least 5 minutes at ambient temperature.
7. Method of any preceding claim, wherein the solution comprises calcium sulfonate in an amount between 1% and 30% by mass.
8. Method of any preceding claim, wherein the solution comprises the hydrophobic wax which is essentially composed of a C20–C35 alkane fraction and calcium sulfonate

9. Method of any preceding claim, wherein the solution further comprises 1,1'-Biphenyl 4,4'-dibromo ($C_{12}H_8Br_2$).

5 10. A steel mechanical part having undergone a surface treatment to improve its resistance to wear and corrosion, comprising a nitrated surface layer impregnated with a hydrophobic wax-based solution, wherein the wax is an organic carbon compound having a molecular weight below 500, and preferably between 200 and 500.

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11. Mechanical part of claim 10, wherein the wax-based layer has a coating weight between 5 and 60 g/m².

12. Mechanical part of any of claims 10–11, wherein the wax-based layer
15 comprises calcium sulfonate in an amount between 1% and 30% by mass

13. Mechanical part of any of claims 10–12, wherein the wax-based solution comprises the wax which is essentially composed of a C20–C35 alkane fraction and calcium sulfonate.