The invention relates to vacuum pump components without conversion layers that are made of valve metals and alloys thereof.
VACUUM PUMP COMPONENTS WITHOUT CONVERSION LAYERS

[0001] The invention relates to vacuum pump components without conversion layers that are made of valve metals and alloys thereof.

[0002] DE 101 63 864 A1 relates to a process for the coating of objects made of valve metals or their alloys with a thin barrier layer consisting of the metal and an oxide ceramic layer provided thereon whose surface has been coated with fluoropolymers, characterized in that the fluoropolymers are introduced into the capillary system of the oxide ceramic layer in the form of a solution by vacuum impregnation, followed by removing the non-wetting portions of the solution and drying. Accordingly, the group of valve metals includes aluminum, magnesium, titanium, niobium or zirconium and their alloys. In addition, this specification also defines further components of vacuum pumps made of valve metals, such as rotors and stators of turbo-molecular pumps.

[0003] In this document and also within the scope of the present invention, “aluminum and its alloys” means ultrapure aluminum and the alloys AlMn; AlMnCu; AlMg, AlMg5; E-ALMgSi; AlMgSi5; AlZnMgCu5; AlZnMgCu5; G-AI5Si5; G-AI5Si5MG; G-AI5Si5Cu5; G-AICu4Ti; G-AICu4TiMg. Further, in addition to pure magnesium, in particular, the magnesium cast alloys with the ASTM designations AS41, AM60, AZ63, AZ81, AZ91, HK31, QE22, ZE41, ZH62, ZK51, ZK61, EZ33, HZ32 and the wrought alloys AZ31, AZ61, AZ80, M1 ZK60, ZK40 are suitable for the purposes of the invention. Further, pure titanium and also titanium alloys such as TiAlV3, TiAlFe2.5 and others may be employed.

[0004] In DE 101 63 864 A1, the oxide ceramic layer is essentially formed by a conversion layer of the surface of the component, so that in practice part of the substrate material is lost and converted to the oxidation barrier layer.

[0005] In addition, it is known that conventional anodization layers, plasma-chemical anodization methods (KEPLA-COAT®) and other methods) are known. Also, it is known to nickel-coat the above mentioned valve metals.

[0006] All the above mentioned coating methods enable true-contour covering surfaces that are possible in known conversion layers, such as KEPLA-COAT®, or anodization layers on the one hand. However, according to the present invention, it is essential that no substrate material is lost by conversion, i.e., no conversion layer is produced. Thus, if needed, the coating can be repeated any number of times without substrate losses, which is highly important for maintenance, in particular.

[0007] Details of process technology can be seen in detail from the above mentioned publications WO 03/029529 A1, WO 2006/047501 A2 and WO 2006/047526 A2. In this respect, these documents are also included herein by reference in their entirety.

[0008] Because of the high deposition rates, the exposure times are reduced to about one third as compared to the usual anodization methods, and even to one sixtieth (quarter) as compared to the above mentioned KEPLA-Coat® method. Thus, a significant economical advantage is provided. In addition, it could be observed that no edge effect has occurred with the vacuum pump components without conversion layers, made of valve metals and their alloys, prepared according to the invention. This property, in particular, has not been known from the above mentioned documents and thus represents a surprising advantage of the present invention.

[0009] As compared to the layers prepared by anodization methods or by the KEPLA Coat® method, a higher resistance to abrasive wear is obtained. The deposited layers may have a hardness of about 700 HV.

[0010] According to the invention, significant advantages in corrosion protection over the known layer systems could be achieved. This includes protection against citric acid and hydrochloric acid, in particular. It is known that anodization layers are sensitive to the action of citric acid, while the KEPLA Coat® layers have no sufficient stability to hydrochloric acid.

[0011] It has been found that the basic service life of the electrolyte can be set by analytical monitoring and optionally replenishing over significantly longer periods than those
US 2014/0154503 A1

required with the previously known methods for the coating of components of vacuum pumps made of valve metals and their alloys. In contrast, the electrolyte of KEPLA Coats® must be discarded depending on usage because of contaminations originating from the starting material. This similarly applies to electrolytes of anodization layers.

[0017] Components of vacuum pumps made of valve metals and their alloys according to the invention include, in particular, rotors, stators, stator disk halves, helical stages, housings and bearing shells.

[0018] In accordance with the prior art, the term “valve metals” herein includes metals of the group of aluminum, magnesium, titanium, niobium and/or zirconium and their alloys. The specific alloys of aluminum, magnesium and titanium as mentioned in the introductory part of the description are also particularly preferred according to the present invention.

[0019] It is particularly preferred to select at least one oxide and/or oxyfluorides of the group consisting of aluminum, titanium and/or zirconium for the surface coating. These are best suitable for realizing the advantages of the present invention.

[0020] In one embodiment of the present invention, the thickness of the surface coating is from 5 to 50 µm. It is particularly preferred according to the present invention that the thickness of the surface coating is from 15 to 50 µm. If the thickness of the surface coating is selected too thin, sufficient protection against corrosion, heat, abrasion and chemicals cannot be ensured. In contrast, if the thickness of the surface coating is selected too large, the corresponding coatings will tend to chip off. In addition, correspondingly thick coatings are economically inefficient.

[0021] Another embodiment of the present invention relates to a process for preparing vacuum pump components without conversion layers, made of valve metals or their alloys, that are produced by electroplating, characterized by:

[0022] (a) providing an anodizing solution containing, in addition to water, at least one other component selected from the group of water-dispersible complex fluorides and oxyfluorides of elements of the group consisting of boron, germanium, aluminum, magnesium, titanium, niobium, hafnium and/or zirconium and mixtures thereof;

[0023] (b) contacting a cathode with said anodizing solution;

[0024] (c) inserting the components as anodes into said anodizing solution; and

[0025] (d) applying a voltage between the anode and cathode to apply a surface coating to said components.

[0026] In principle, this process is already known from the mentioned documents WO 03/029529 A1, WO 2006/047501 A2 and WO 2006/047526 A2. The present invention is distinguished therefrom by the selected components of vacuum pumps made of valve metals and their alloys.

EXAMPLES

Example 1

[0027] A sample sheet of AlMgSi, with dimensions of 100x50x1.5 mm was subjected to anodic coating at 400 volts for 5 minutes in an electrolyte as described in WO 03/029529 A1, WO 2006/047501 A2 and WO 2006/047526 A2 within 5 minutes. The determined layer thickness was about 10 µm.

Example 2

[0028] A sample sheet as described in Example 1 was coated in an analogous way within 10 minutes. The determined layer thickness was about 12 µm.

Example 3

[0029] The sample sheets coated according to Examples 1 and 2 were exposed to a hydrochloric acid atmosphere formed above a bath containing 15% by weight hydrochloric acid. The oxide ceramic layer on the sample sheets was examined for chipping off after test durations of 144 hours and 300 hours. The oxide ceramic layer on the sample sheets was still intact after this exposure time.

Example 4

[0030] The sample sheets coated according to Examples 1 and 2 were exposed to citric acid solutions having concentrations of 2%, 3.5% and 5%. The oxide ceramic layer on the sample sheets was examined for chipping off after a test duration of 90 hours. The oxide ceramic layer on the sample sheets was still intact after this exposure time.

1. Vacuum pump components without conversion layers, made of valve metals or their alloys, characterized in that their surfaces have a coating of at least one oxide and/or oxyfluoride of an element of the group consisting of boron, germanium, aluminum, magnesium, titanium, niobium, hafnium and/or zirconium and mixtures thereof, produced by electroplating and having a layer thickness within a range of from 5 to 50 µm.

2. The components according to claim 1, including rotors, stators, stator disk halves, helical stages, housings and bearing shells.

3. The components according to claim 1, characterized in that said valve metal is selected from aluminum, magnesium, titanium, niobium and/or zirconium and their alloys.

4. The components according to claim 1, characterized in that said coating consists of at least one oxide and/or oxyfluoride of the group consisting of aluminum, titanium and/or zirconium.

5. The components according to claim 1, characterized in that the thickness of the surface coating is from 15 to 30 µm.

6. A process for preparing vacuum pump components without conversion layers, made of valve metals or their alloys, that are produced by electroplating, characterized by:

(a) providing an anodizing solution containing, in addition to water, at least one other component selected from the group of water-dispersible complex fluorides and oxyfluorides of elements of the group consisting of boron, germanium, aluminum, magnesium, titanium, niobium, hafnium and/or zirconium and mixtures thereof;

(b) contacting a cathode with said anodizing solution;

(c) inserting the components as anodes into said anodizing solution;

(d) applying a voltage between the anode and cathode to apply a surface coating to said components.

* * * * *