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INVESTIGATION OF THE OPERATIONAL CHARACTERISTICS OF PLASMA-CHEMICAL STRENGTHENING THIN-FILM SILICON-CONTAINING COATINGS, DEPOSITED FROM ARC PLASMA AT ATMOSPHERIC PRESSURE

A. L. Halazubau

Candidate of Technical Sciences, Associate Professor, Associate Professor of the Department of Engineering and Pedagogical Education, Mozyr State Pedagogical University named after I. P.Shamyakin, Mozyr, Belarus, e-mail: golozuboval@ya.ru

Abstract

The strengthening of metal surfaces by applying thin-film silicon-containing coatings deposited from arc plasma at atmospheric pressure allows them to be protected from wear and aggressive environments, and is ultimately determined by the performance characteristics of such coatings.

One of the basic operating performances of hardening protective thin-film coverages is their thickness. Definition of thickness of thin films by direct gauging represents an intractable technical problem.

In the paper, the use of an optical interference picture in the system of a substructure thin-film coverage for determining thickness is offered. The given approach allows determining and controlling the coating thickness not only on completed product but also during the coating process. An experimental study of the thickness of a thin-film silicon-containing coating using a scanning electron microscope showed that the film thickness is within 1,1–1,2 μm. Comparison of the calculated and experimental data on the thickness of the strengthening coating showed high convergence of the results (12–15 %). The ability to precisely control the thickness significantly expands the scope of application of thin-film strengthening silicon-containing coatings applied from argon arc plasma at atmospheric pressure.

Precise control over coating thickness significantly expands the range of applications for thin-film silicon containing strengthening coatings deposited from arc argon plasma at atmospheric pressure.

The results obtained during theoretical and laboratory studies were used in real production conditions at the largest enterprise of the Republic of Belarus, Open Society Mozyr Oil Refinery. In the conditions of repair and mechanical production, an author's installation for applying thin-film coatings and a technological process for applying corrosion-resistant thin-film silicon-containing coatings on parts operating under conditions of non-abrasive wear and in contact with aggressive environments were introduced.

Keywords: hardening, a thin-film siliceous coverage, arc plasma, thickness, an interference.

ИССЛЕДОВАНИЕ ЭКСПЛУАТАЦИОННЫХ ХАРАКТЕРИСТИК ПЛАЗМОХИМИЧЕСКИХ УПРОЧНЯЮЩИХ ТОНКОПЛЕНОЧНЫХ КРЕМНИЙСОДЕРЖАЩИХ ПОКРЫТИЙ, ОСАЖДАЕМЫХ ИЗ ДУГОВОЙ ПЛАЗМЫ ПРИ АТМОСФЕРНОМ ДАВЛЕНИИ

А. Л. Голозубов

Реферат

Упрочнение металлических поверхностей нанесением тонкопленочных кремний содержащих покрытий, осаждаемых из дуговой плазмы при атмосферном давлении, позволяет защитить их от износа и действия агрессивных сред и в конечном счете определяется эксплуатационными характеристиками таких покрытий.

Одной из основных эксплуатационных характеристик упрочняющих защитных тонкопленочных покрытий является их толщина. Определение толщины тонких пленок непосредственным измерением представляет собой трудно разрешимую техническую задачу.

В статье предлагается использование оптической интерференционной картины в системе подложка – тонкопленочное покрытие для определения его толщины. Данный подход позволяет определять и контролировать толщину покрытия не только на готовом изделии, но и непосредственно во время нанесения. Экспериментальное исследование толщины тонкопленочного кремний содержащего покрытия с помощью растрового электронного микроскопа показали, что толщина пленки находится в пределах 1,1–1,2 мкм. Сопоставление расчетных и экспериментальных данных о толщине упрочняющего покрытия дали высокую сходимость результатов (12–15 %).

Возможность точного контроля толщины значительно расширяет область использования тонкопленочных, упрочняющих, кремнийсодержащих покрытий, наносимых из дуговой аргоновой плазмы при атмосферном давлении.

Полученные в ходе теоретических и лабораторных исследований результаты были использованы в условиях реального производства на крупнейшем предприятии Республики Беларусь ОАО «Мозырский нефтеперерабатывающий завод». В условиях ремонтно-механического производства была внедрена авторская установка для нанесения тонкопленочных покрытий и технологический процесс нанесения коррозионно-стойких, тонкопленочных, кремнийсодержащих покрытий на детали, работающих в условиях неабразивного изнашивания и в контакте с агрессивными средами.

Ключевые слова: упрочнение, тонкопленочное, кремнийсодержащее покрытие, дуговая плазма, толщина, интерференция.

Introduction

Drawing of thin-film siliceous coverages from arc plasma at atmospheric pressure is new enough direction in a work-hardening range of details of cars [1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13]. Working capacity of thin films is influenced by variety of factors – cohesive and adhesive properties of a coverage, and also property of a metal substructure [8, 14, 15, 16].

Hardening of metal surfaces by drawing thin-film silicium of containing coverages, besieged of arc plasma at atmospheric pressure allows to protect them from deterioration and operation of excited environments and eventually is defined by operating performances thin-film silicium of containing coverages. Definition of operating performances is necessary and a sufficient condition for an estimation working capacity and longevity of system «a substructure - a coverage». Those are: hardness (microhardness), adhesion, phase and a chemical compound, wear resistance. Important performance of a sheeting is also it thickness which can influence considerably on its operation features, defining including its longevity [17, 18].

Definition of thickness thickness of a thin-film coverage

Samples from fragments thin-film silicium of containing coverages separated from a substructure were examined by the RSMA-ANALYSIS method on REM "Nanolab-7" with a spectrum of power variance EDS "Sistem 860".

As a result for films on the basis of a silicon oxide the following data on chemical element composition has been obtained: Si – 60,84 %, O – 39,02 %, C – 0,14 %. For films on the basis of silicium carbide: Si – 73,22 %, C – 26,7 %, O – 0,08 %. For films on the basis of silicium nitride: Si – 67,72 %, N – 32,2 %, O – 0,08 % [16, 18, 19].

Thus the conducted probes allow to make conclusion that received of the arc plasma containing products pyrolysis silicium of the organic, hardening siliceous thin-film silicium of containing coverages represents a difficult pseudo-alloy not stoichiometric composition, containing in the composition oxides, carbides and the silicium nitrides, not having a crystalline structure and representing uncrystalline (passer-by on the glass) structure. The obtained data will agree with results of probes [3, 8, 11, 20] also specifying in uncrystalline structure of the thin films received by sedimentation from ion bundles in vacuum.

Ratio change between components in thin-film silicium of containing coverages is possible at the expense of a variation of composition of arc plasma (a gas phase) a path the additional introductions of elements and joints in necessary densities, and also at the expense of process control in temperature the purposeful the job of its parametres [6, 8, 9, 21].

The picture observed in an optical microscope confirms an optical transparency hardening thin-film silicium of containing coverages (Figure 1).

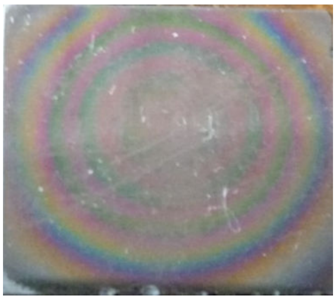

Figure 1 – The Sample with plotted thin-film silicium of containing coverages. Material of substructure WC-Co, the polished surface

At small thickness of transparent films in an optical range, as the most exact and simple method of study of their shape and thickness serves visual observation changes of an interference picture on a surface of the substrate with simultaneous timekeeping [5, 18, 22].

For growth kinetics probe samples with the polished a surface for deriving of an accurate optical picture were used at when applying thin-film silicium of containing coverages [7, 18, 23, 26].

In the course of drawing plasma of the chemical coverages at stationary position plasma the generator (plasmatron) on the processed surfaces series of the optical phenomena is observed. In the beginning handlings, the polished surface of the sample starts to lose gradually a reflecting capacity, acquiring dark colour. Further handling leads to colour change on dark blue then in the hcentre of a plasma spot start to appear sequentially the expanding circles of the colours making a spectrum of a natural light. Circles grow to certain magnitude (diameter of 6–10 mm) then in the centre the monochrome a circle there is a growing circle of following colour. In result on a surface the picture of the concentric colour rings similar to rings of Newton is observed.

By results of the experiment, shown in table 1 the graph of dependence of thickness of a besieged coverage from duration of handling of a substructure which is presented in Figure 2 is constructed.

On a curve there are some characteristic sites, the corresponding to different stages of formation and coverage growth.

Table 1 – Dependence of occurrence of maxima on duration of handling by a plasma stream

Appearance a colour picture	Experiment number (time in seconds)				
	1	2	3	4	5
Dark-blue	16	17	16	12	14
1 red	26	33	30	29	31
2 red	41	48	40	48	46
3 red	63	72	62	75	64
4 red	92	95	90	91	94
5 red	120	115	117	122	116

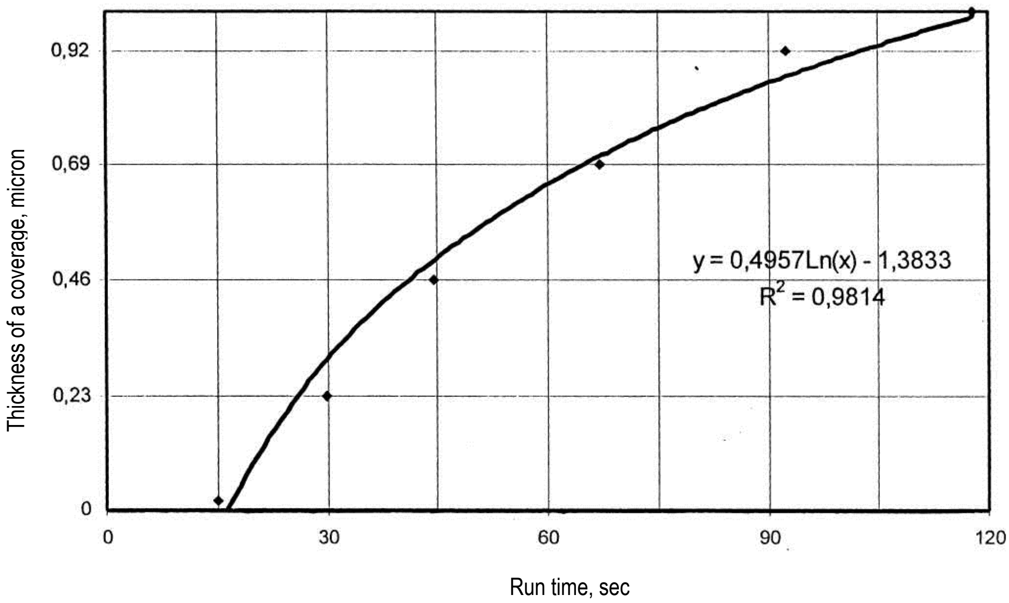

Figure 2 – Growth kinetics thin-film silicium of containing coverages

Experiments by coating separation from a substructure have displayed that the coverage is optically transparent, and its thickness decreases from the centre to periphery, according to radial distribution of temperatures in plasma the jet [22, 24]. Such in the image, the observable optical picture grows out interference of light in system a thin lens-metal a substructure. In this case, from geometrical reasons (Figure 3), we have

$$r_k^2 = R^2 - (R - h_k)^2 . \quad (1)$$

where r_k – radius of k-rings, R – radius of curvature of a lens, h_k – height of a lens from a surface of a coverage to a plain of k-rings.

After transformations with the account that $h_k^2 \to 0$, we have

$$r_k^2 = 2 \cdot R \cdot h_k\ , \tag{2}$$

accordingly

$$r_{max}^2 = 2 \cdot R \cdot d\ , \tag{3}$$

where r_{max} – radius of the maximum ring.

R – radius of curvature of a lens, h_k – height of a lens from a surface of a coverage to a plain of k-rings, d – thickness of a lens (film) on the centre.

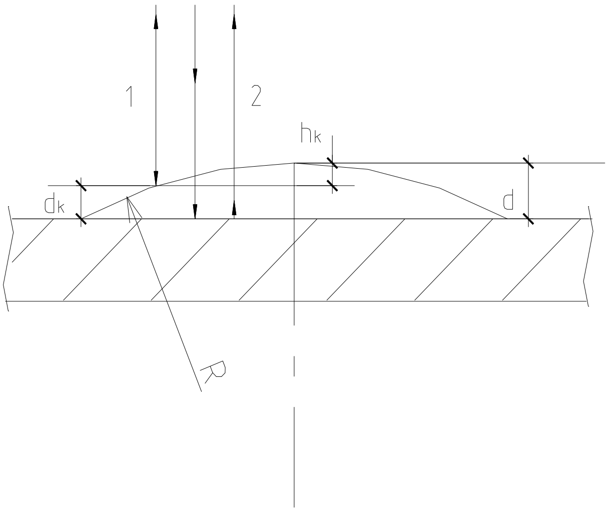

Figure 3 – The Settlement circuit of definition of thickness thin-film silicium of containing coverages

Considering that both rays are reflected from optically more tight environments (in the first case from a lens material, in the second from a surface metal) with phase loss on half of wavelength in each case, condition a maximum will be recorded in an aspect

$$d = 2 \cdot d_k \cdot n = k \cdot \lambda, \tag{4}$$

where n – an index of refraction of a lens (film), (k = 0,1, 2...).

From here

$$d_k = \frac{k \cdot \lambda}{2 \cdot n}. \tag{5}$$

Considering that $d_k = R – h_k$, we will receive

$$h_k = R - \frac{k \cdot \lambda}{2 \cdot n}. \tag{6}$$

Substituting (2) in (1) it is had

$$r_k^2 = 2 \cdot R^2 - \frac{(R \cdot \lambda \cdot k)}{n}. \tag{7}$$

From here, having expressed a lens radius of curvature through radius of i and k rings and taking into account (1) we receive expression for definition thicknesss coverages

$$d = \frac{r_{max}^2 \cdot (i-k) \cdot \lambda}{2 \cdot (r_i^2 - r_k^2) \cdot n}. \tag{8}$$

As a result of gaugings next sizes of radiuses of rings have been defined: r_{max} = 9,1 mm, r_i = 7,0 mm, r_k = 5,5 mm. The index refractions for thin-film silicium of containing coverages composition SiO_2 is equal n = 1,458 ± 0,002 [25]. The wavelength of light corresponding to red colour is accepted equal 672 nanometers. Thus, after evaluations under the formula (8) it is had maximum thickness lenses

$$d = 1,017 \pm 0,092\ \mu m.$$

Thickness change coverages, leading to changeover of a colour gamma, it is possible to define thickness change from the output before regularities. Distance (thickness of a film) between the next maxima on a surface (rings of one colour) in conformity about figure 1 are defined on ratio

$$h_k - h_i = \frac{i \cdot \lambda}{2 \cdot n} - \frac{k \cdot \lambda}{2 \cdot n} \tag{9}$$

$$\Delta h = \frac{\lambda}{2 \cdot n} = \frac{672 \cdot 10^{-9}}{2 \cdot 1,4584} \approx 0,23\ \mu m.$$

Thus, occurrence of a following ring of identical colour in process processing testifies to increase in thickness of a film at 0,23 microns.

It allows operatively and to watch precisely enough thickness of a plotted coverage in the course of handling that is important parametre of technique. Besides, the test coating on substructures allows to establish univalent connection with simultaneous timekeeping between thickness of a coverage and a run time for the given mode of behaviour of installation. It does possible to realise exact control of thickness thin-film silicium of containing coverages in places where direct observation of an interference picture, for example, in holes of small diameter through passage, including, die holes is impossible. Feature of drawing thin-film silicium of containing coverages from arc plasma at atmospheric pressure (as transport response), is ability of drawing of coverages in holes of small diameter through passage, and also under any edges to a shaft of a plasma stream, thus exact control of thickness of plotted coverage thin-film silicium of containing coverages allows to harden such difficult details with a thickness quality assurance that cannot achieve with use of traditional techniques.

For experimental definition of thickness thin-film silicium of containing coverages it was used REM "Nanolab-7" and a fragment of the coverage separated from a substructure. Gaugings on a back of the sample have displayed that thickness of a film is in within the limits of 1,1–1,2 microns [14, 18]. Thus, for the first time compared settlement and experimental data about thickness hardening thin-film silicium of containing coverages, received by sedimentation from arc plasma, have given high convergence of results (12–15 %). Toe-outs in values most likely are linked with an aberration of curvilinear external surface thin-film silicium of containing coverages from a theoretical surface of a thin lens with fixed radius.

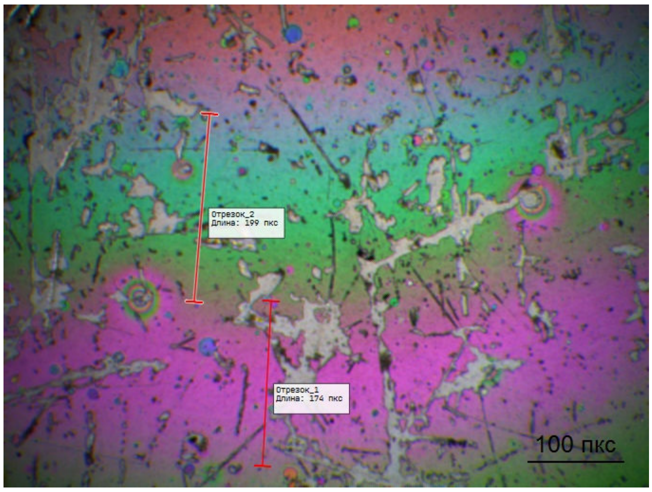

Figure 4 – Color gamma on surface thin-film silicium of containing coverages. An optical microscope «Altami» with a digital camera. 1 pixel = 3,2 × 3,2 a micron

Exact values of thickness allow to delineate area of use coverages, in particular, obviously that the coating thick to 0,3 microns gets to a zone of the tolerance for all qualifications exactitude and consequently process on a coating can be finishing (not demanding aftertreatment).

The maximum thickness besieged thin-film silicium of containing coverages has made 2 microns. Receiving such thicknesss it has appeared possible only at use special the measures preventing overheating of a substructure. Thus processing it was carried on by cycles till 15–20 seconds, with an interval 20–30 seconds, and with insignificant lowering

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(on 20–30 %) densities of metalloorganic joint in plasma after application coverages thickness more than 1 microns. However, spent tribological trials have displayed that in the best performances possess thin-film silicium of containing coverages thickness from 0,7 microns to 1,0 microns.

Thus, having possibility of exact control of thickness of a coverage, we can essentially expand area of use of technique. Control of thickness of a coverage on a colour gamma allows to plot thin-film silicium of containing coverages the set thickness on surfaces of any configuration and shapes. And there is possible drawing thin-film silicium of containing coverages of the set thickness on local sites, i.e. for example strips of the set thickness and length. Figure 4 also there is possible drawing thin-film silicium of containing coverages of identical thickness, including on a surface of a difficult configuration that is almost impossible with use of traditional methods of drawing of protective hardening coverages. It is necessary to mark that occurrence of an interference picture in a place of drawing thin-film silicium of containing coverages takes place not only for the polished surfaces, but also for surfaces after a coarse finishing, for example polishings, millings, turning. In this case the picture interference rings becomes more diffusion and not having clear boundary, but nevertheless allowing to define thickness with enough split-hair accuracy (to 20 %).

The observable picture of interference rings (Figure 1) is characteristic only for a fixed plasma stream, i.e. at exhibiting plasmatron over one point. The longitudinal travel plasmatron allows to plot thin-film silicium of containing coverages in the form of strips of the set thickness (Figure 5).

In the conditions of real manufacture most often there are situations when it is necessary to harden surface of revolutions (shafts, axis). In this case shaft rotation is carried out in accommodating, and the coating on extended sites occurs for the movement score plasmatron along a shaft shaft therefore the coverage is plotted on extended sites.

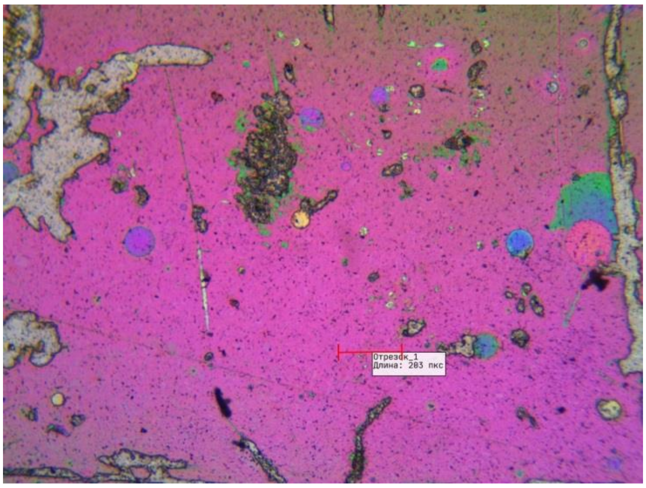

Figure 5 – A site of a surface with identical thickness thin-film silicium of containing coverages

Received during theoretical and laboratory probes results have been used in the conditions of real manufacture at the largest operation of republic of Belarus Open Society Mozyr Oil Refinery. In the conditions of mechanical-repair manufacture author's installation for drawing of thin-film coverages and a master schedule of drawing of noncorrosive thin-film siliceous coverages on a detail, working in the conditions of not abrasive wear process and in contact to excited environments has been introduced. Surfaces of shafts of pumps of centrifugal type PC (a material-steel 40X, 290 HB) intended for pumping the oil, the liquefied hydrocarbon gases and oil products were hardened at temperature of a pumped over fluid from 193 K (–80° C) to 673 K (+400° C) with viscosity from $0{,}5 \cdot 10^{-4}$ m^2/sec to $8{,}5 \cdot 10^{-4}$ m^2/sec.

Principal causes of outage of shafts of pumps were: excess of supposed escape through shaft seals and deterioration of mounting surfaces to pinion point under bearings and he impeller. The given problem has been solved for the score plasmochemical drawings of its – thin-film sheetings saving resources the technique allowing safely to protect metals from staining and deterioration by drawing on their surface of chemically steady siliceous joints, having high physicomechanical properties («Anticorrosive guard of details of the nasosno-compressor equipment» certificate of registration № 2003536, «Working out of a master schedule of hardening and a corrosion prevention of seats of bearings and face seals» certificate of registration № 20023233).

Thus thin-film silicium of containing coverages it was used not only in the form of a noncorrosive coverage, but also as a barrier coverage for separation of contacting mounting surfaces to pinion point under bearings and impeller, at joint assembly-demolition that has led to change of conditions of deterioration yo-to reduction or complete an exception microcutting at the expense of surface hardness increase. In this case the optimum condition-elastic interaction of conjugated surfaces is ensured.

Operational trials of shafts of pumps of centrifugal type PC (cantilever) for pumping oil products in the conditions of real manufacture have displayed that the period of their use was augmented in 2,5–3 times at the expense of an exception of effect of the active corrosion environment and deterioration by mounting surfaces to pinion point of shafts that has allowed to cut expenditures on reconditioning of the equipment and to raise its reliability.

Conclusion

The method of definition used in operation on an interference picture of thickness of the protective hardening thin-film siliceous coverages besieged from arc plasma at atmospheric pressure, has displayed split-hair accuracy with simultaneous possibility not only definitive, but also intermediate and a process control inspection.

Reliability of the received theoretical dependences is confirmed by experimental researches of the separated fragments of a coverage methods of scanning electron microscopy.

The obtained data about thickness thin-film silicium of containing coverages allows to define and specify areas of use of such coverages in various areas of engineering.

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