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Correlation of the Microrelief of the Surface of Structure and Magnetic Properties of Oxidic Coverings on the Titan

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Abstract. The technique of system parametrization of a microrelief of surfaces plasma electrolytic oxidation coverings on the titan is offered. Correlation of topology of surfaces of oxidic coverings with their thickness and magnetic properties is established.

Introduction

Functional coatings are perspective as materials that absorb electromagnetic radiation, also as magnetic switches and mikrotransformers. In some cases, for example, in matters of adsorption, catalysis, triboengineering, the decisive role played by statistics microrelief. We emphasize that these micro materials have extremely high topological, morphological complexity, and represent a holistic system of inhomogeneties without the possibility of their componential release. In this situation, it is logical to refer to the method that does not involve component-selection procedures, and not even attract decomposition methods. On the other hand these characteristics allow us to connect micro surface coatings with conditions for obtaining, recording, etc. properties. We have studied the micro surface oxide coatings, formed by plasma electrolytic oxidation (PEO) in the electrolyte-suspension Na_3PO_4+ $Na_2B_4O_7$+Na_2WO_4+$Fe_2(C_2O_4)_3$ on titanium at different current densities i. Micrographs of coatings (Fig. 1) was obtained on a raster electron microscope in the regime of secondary electrons, that means the topography of the surface of the coating was observed. Were chosen five conditions of formation of coatings depending on the amount of missed electricity $Q = i \cdot t$, where i - current density formation in A/cm^2, t - formation time in seconds (Q = 12, 30, 60, 90, 120 C/cm^2) Magnetic measurements were made on 7 SQUID MPMS magnetometer at temperatures ranging from 300 to 2 K. In the calculation of the magnetization measured magnetic moment normalized by the mass of the sample with the coating. Measuring the thickness of coatings were made by eddy current thickness calipers “BT-201”.

Methodic

Basic numerical analysis technique of the microrelief is based on calculating Fraunhofer diffraction patterns (FDP) by digital method [1]. Typical FDP received from the interfaces of the samples are shown in Fig. 1. The nature of the defect distribution of the microrelief studied surface appears in the degree of anisotropy FDP. Energy concentrated in different parts of the Fourier spectra, reflects the characteristics of various irregularities present in the image. In order to introduce a relatively simple parameterization statistics of the microrelief surfaces and develop physical and mathematical foundations of rapid methods of the statistical analysis that allows to track and compare modes, an invariant transformation FDP into an integrated frequency response (IFR) is made. Using the procedure described in [2], first translate the IFR in the space of integral functions of Lebesgue measure (IFML) μ(x), then we get a measure of similarity- difference statistics of the microrelief surfaces from a base distribution $\tilde{\mu}(x)$. In its mathematical sense, this measure is linearized divergence Kullback - Liv I.

$$\mathbf{LivI}\left[\frac{\mu(\mathbf{x}_i)}{\tilde{\mu}(\mathbf{x}_i)}\right] = \sum_{\{x_i\}} \frac{|\mu(\mathbf{x}_i) - \tilde{\mu}(\mathbf{x}_i)|}{\tilde{\mu}(\mathbf{x}_i)},$$

where i varies from 1 to N - the number of quantization levels of IFR, in our case - 40. It is easy to show that it has all the properties of a metric, and not just in the case of distance Kullback divergence [4]. Error in the assessment of L-divergence in our data: N = 40, $|\delta\mu(\mathbf{x})| = 0.1\langle\mu(\mathbf{x})\rangle$, $\langle\mu(\mathbf{x})\rangle = 0.4$; is given by:

$$\Delta\,\mathbf{LivI}\left[\frac{\mu(\mathbf{x})}{\langle\mu(\mathbf{x})\rangle}\right] = \frac{|\delta\mu(\mathbf{x})|}{\sqrt{N}\,\langle\mu(\mathbf{x})\rangle^2} = \mathbf{0.04}\ ,$$

upon application of the normal noise from 25% var.

The proposed mathematical model of describing the spectra of irregularities that enable you to enter the measurement by comparing the spectral estimates, bypassing the stage of pre-filtering of random realizations of the microrelief. There is no need to establish a priori unknown composition law, which determines the type of algorithms for detecting simple components, which are the object of analysis in the usual mathematical statistics of random processes. This is the systemic, holistic approach to the problem of identification of complex spectra of spatial inhomogeneities.

Results

Even at the level of visual image analysis (Fig. 1) it is easy to see that the surface microrelief is significantly different for the different modes of receipt. In Fig. 1a shows the initial period of the coating (Q = 12 C/cm^2) which is based on the of the microrelief of the substrate. On FDP to a wide spectrum of inhomogeneities in the range 1 ÷ ~ 100 microns is presented. In the next phase (Q = 30 C/cm^2) begins the coating, but it is not completed in full, and has the character of elongated "islands" with a size of less than about 10 microns. Next (Q = 60 C/cm^2, Fig. 1b) islands combine to form a continuous grid of bumps and hollows. On FDP this is reflected in further narrowing of the spectrum. The following mode (Q = 90 C/cm^2) is characterized by the disappearance of the brokenness of the microrelief, which becomes more "smooth", without abrupt changes but appears an anisotropy in the distribution of defects on the surface, as evidenced by the elongation (ellipsoidal shape) of FDP. This may be due to the anisotropy of the pore distribution. Last mode (Q = 120 C/cm^2, Fig. 1c) does not make any visible changes in the microrelief itself or in the distribution of irregularities on the surface.

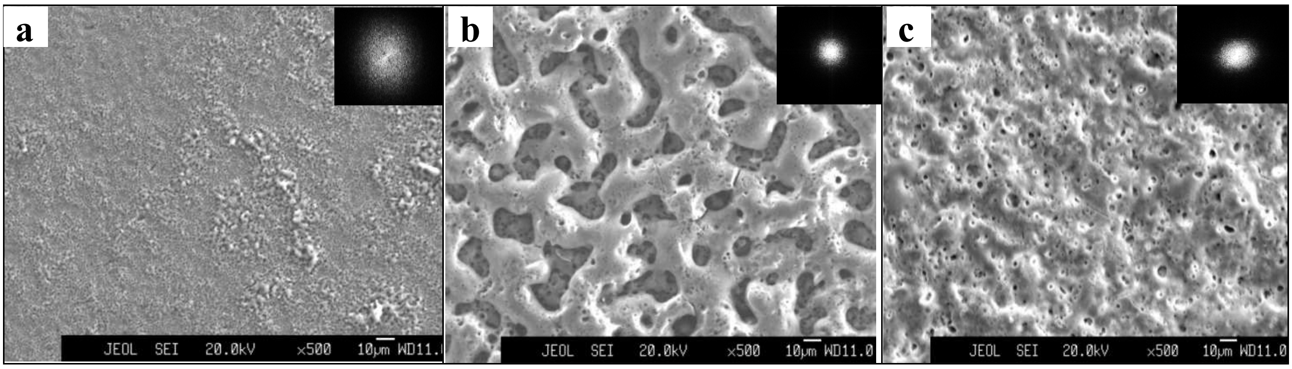

Fig. 1 - Microrelief surface oxidized coatings on titanium produced by the amount of missed electricity (a) - 12 C/cm^2, (b) - 60 C/cm^2, (c) - 120 C/cm^2 and corresponding to them of FDP.

In Fig. 2 shows a comparison of statistics of the microrelief. In Fig. 2a for comparison base taken a spectral estimate S(k), which has a linear IFML that leads to $\frac{dS(k)}{dk} = const$ - white evaluation (noise) of the derivative of S(k), i.e. the rate of change in intensity throughout the k-scale is the same (linear envelope). This approximation shows that the overall picture of the behavior of the microrelief regime falls with Q = 12 C/cm^2. To keep track of the more subtle changes for the other four modes of its IFML and was taken as basis for comparison. This result is shown in Fig. 2b. You can see that the changes in the surface microrelief continue and dependence Liv I (Q) has a minimum, i.e. we can say, in a sense of "optimal" mode. The question is, what are the differences of surfaces and for which tasks fit a particular type of surface will require of an additional study for a particular process. Apparently, there is a threshold regime, after which a further increase of Q increases the variability of microrelief, bringing together the regimes at Q = 30 - 60 C/cm^2 and Q = 120 C/cm^2 on statistics of the microrelief [3].

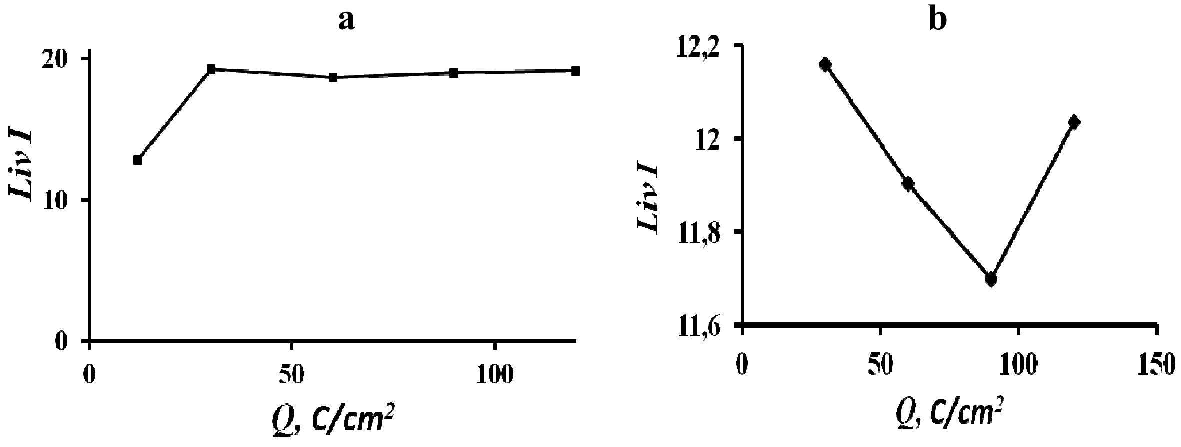

Fig. 2 - Linearized Kullback divergence on Lebesgue measures of spectral estimates of the microrelief oxidized coatings. Taken as the base of comparison: (a) - IFML in a straight line approximation, (b) - IFML mode Q = 12 C/cm^2.

To estimate the correlation of changes in surface microrelief with the physical characteristics of the objects were measured the coercive force and the thickness of the coating, depending on the modes of production. These results are presented in Fig. 3.

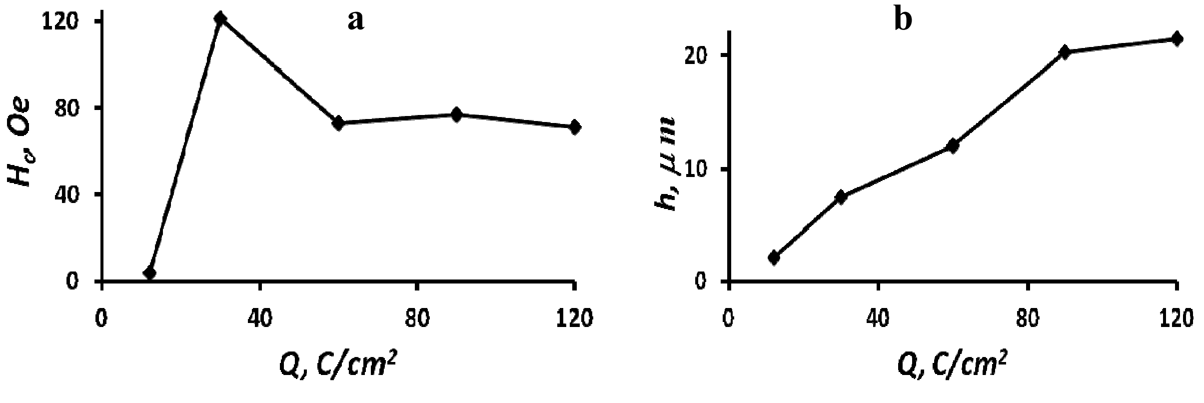

Fig. 3 Influence of the number of missed electricity Q coercive force Hc (a) and the thickness h (b) of coatings.

Mutual analysis of Fig. 2 and Fig. 3 allows you to select mode with Q = 30 C/cm^2 by precipi-shaped behavior of the coercive force (Hc = 121 Oe). Even under high Q, it is at a level ~ 70 Oe. This behavior Hc is explained by the fact that in the coatings with h ~ 7 mm (Fig. 3b) there are the crystalls that are responsible for the ferromagnetic properties which in the pores are small. With the growth of pore size (with Q ~ 30 C/cm^2) and, accordingly, the crystallites, the transition from the their superparamagnetic state to single-domain (for a maximum of the curve) and, further, to a multi-domain [5]. Naturally, such a transition cannot be sharp because of the existence of a broad distribution of crystallites in size. Last causes the presence of the distribution of energy barriers, overcoming delayed in relation to the external magnetic field which is one of the reasons for the hysteretic behavior of the magnetization. Coercive force will also depend heavily on the relaxation of the magnetization of the crystallites, which in general obeys the exponential law [6], i.e. depend on the frequency of the alternating magnetic field.

Thus, in order to get the most Hc it is necessary to stop- process of coating formation in the early stages, to a certain value (in the Q = 30 - 60 C/cm^2). This is important, because of relatively thin coating that provides high adhesion to metal, resistance to deformation, lower porosity, optimizes energy consumption in obtaining magnetically metal oxide systems by PEO.

Comparison of Fig. 3b and Fig. 2b shows that increasing the thickness of the Q = 90 to 120 C/cm^2 is quite weak, the process is almost saturation. But variations of microrelief is changing Liv (Q) (Fig. 2b) begins to increase, passing through a minimum of its value. Such behavior is due to the fact that trends are changing in the process of forming of the coating. The mechanism of oxidation at this stage has much impact on the already existing structure than on the building new one. Consequently, the surface morphology of the coating is changing. It is in the vicinity of Q = 90 mode C/cm^2 surface pores are filled with iron elements [3], which in turn changes the statistics of microrelief (Fig. 2b).

Determining to what purpose (e.g., catalysis or adhesion) is more suitable one or another type of surface will require additional research. But this methodic allows without additional installations, based only on the statistics of the microrelief, get close to the required samples (reference). It is worth mentioning that all of this can be done in real time, with the use of digital media processing.

Conclusion

Thus, using the proposed method of parameterization of surface microrelief of oxidized coatings can knowingly choose in behalf their mode of formation of the purposeful obtaining desired properties of the surface. This approach in future might create structural and morphological passports of obtained coatings.

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