Experimental and theoretical study of the hydriding behaviour in the pulse ecm of titanium alloys

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Abstract. The titanium alloy hydriding is a negative factor since it may result in a slow-action destruction of parts operating at small loads. The introduction of pulse electrochemical machining requires consideration and evaluation of this factor’s influence onto the operating performance of components. Since electrochemical machining is performed at small gaps, and hydrogen release is very intensive on electrodes, favourable conditions for the hydriding process are developed. The work describes a profound theoretical study of this process with proposing technological methods to reduce hydriding.

1. Introduction

An important task of the aircraft engine building is enhancing its competitive ability by virtue of the improved quality, operational reliability and extended service life along with reducing the self-cost of the major engine components. These components conditioning the level of the engine building product are primarily the compressor and turbine blades. Solving this task as applied to these components is possible within the framework of the finishing improvement.

One of the methods in improving the finishing machining of compressor blades is the use of pulse electrochemical machining (ECM) at a final stage as the most appropriate finishing airfoil forming method. Minimal power and thermal effect on the low-hardness airfoil is ensured by the ECM when removing the stock, which is especially important in the absence of the developed technological bases, a small stock and its considerable irregularity. The introduction of ECM is restricted by the technological heredity factors, which partially impair the surface layer condition, for example, by hydriding the machined surface of a titanium-alloy blade airfoil.

It is obvious that when using the electrochemical pulse dimensional machining (ECM) of the blade airfoil as a finishing forming operation, the importance of hydriding as a negative technological heredity factor will be considerably intensified. This is due to the fact that the surface layer formed in the ECM will be definitive of the component’s service life.

The reason of surface hydriding is high diffusion mobility of hydrogen atoms, which is conditioned by its small size. The problems of hydrogen interaction with metals are diverse and they include adsorption, diffusion, dissolution and formation of hydrides. In addition, the negative impact of components’ surface hydriding affects their operational performance as a result of the hydrogen brittleness.

2. Materials and Methods

Surface hydriding occurs in various engineering processes and is the subject of the detailed study. So,
in work [1], when studying the titanium alloy laser welding process, it was detected that if the hydrogen concentration is exceeded by more than 0.2%, the superplastic ductility of the welded joint will decrease, the deformation ratio non-uniformity will grow. In article [2], when studying the stability of Ti₃SiC₂ base plate in hydrogen at the temperature of 1200–1400°C during three hours, the hydrogenation mechanism using the combined use of x-ray diffraction, scanning electron microscopy and the diffused light spectrometry was clarified. The authors observed separation of the base plate layer at the temperature of above 1300°C due to the increased hydrogen diffusion along the basal plane. In work [3], the impact of hydrogen on the structural and phase state of the fine-grained and submicrocrystalline two-phase Ti-6Al-4V titanium alloy was studied by electronic microscopy and by the x-ray diffraction analysis. It was established that hydriding results in minor structural and phase changes both in the fine-grained and submicrocrystalline samples. The use of electron-beam irradiation combined with heating for hydrogen release in Ti-6Al-4V alloy resulted in the decreased degasation time and the reduced hydrogen concentration down to the values adopted in the engineering standards for the Ti-6Al-4V alloy.

In work [4], comparative studies of the effect caused by 0.002–0.12 % hydriding on the structure and phase composition of the submicrocrystalline and coarse-grained Ti-6Al-4V alloy were described. The evolution of deformation processes in hydrogenated alloys within the extension conditions at the temperature of 293 K was studied depending on the hydrogen content and the condition of alloy. The presence of hydrogen in solid-solution Ti-6Al-4V nanostructured alloys was established, which resulted in their reduced yield as well as the improved tensile strength.

Work [5] studied the peculiarities of the ultrafine-grained structure formed in the Ti-6Al-4V alloy through the use of the method combining the intensive plastic deformation and the reversible hydrogen treatment. It is shown that martensitic and α→β phase transformations, due to the presence of hydrogen, favour the improved structure during the intensive plastic deformation of the alloy. This allows reducing the deformation rate necessary to obtain the ultrafine-grained structures with the component dimensions of 0.1–0.5 μm. It was demonstrated that the formation of ultrafine-grained structures in the Ti-6Al-4V alloy results in the growth of its strength properties of about 1.5 times, the increase of hydrogen brittleness resistance and the reduction of superplastic condition transition temperature by 200-300 K.

In article [6], thermo-emf and the electrical resistivity of hydrogenated titanium were measured. Furthermore, the hydrogen concentration in titanium has changed from 0.005 to 0.25 %. The methods applicable to their sensitivity to thermo-emf and electrical resistivity change during the titanium hydrogenation process were compared. Precision of the results obtained was checked by comparing them with the results for palladium. It was shown that the specified method is sensitive to hydrogen concentration in titanium at different alloy hydrogenation depths. Based on the measurements carried out, the authors proposed a graphic method for detecting the titanium structure change.

A large number of works are devoted to the issue of reducing hydrogenation during the titanium alloy ECM, where authors attempted to find the best composition of electrolyte and selecting the optimal machining mode capable of ensuring the low surface roughness value and the small surface layer hydrogen concentration along with the high machining precision. So, for example, the compositions of electrolytes were developed, where the surface smoothing effect is achieved at regular as well as at low current densities. According to the data [7], with the standard ECM conditions, high process performance is achieved in chloride-nitrate electrolyte. For the chloride-bromide electrolyte, the reduced hydrogenation level is observed in proportion to the decreasing surface roughness.

The author selected the optimal electrolyte for the VT3-1 alloy. Mixture 14% NaCl+11% NH₄NO₃ was preferred, where the metal removal rate was 0.85 g/min instead of 0.7 for the sodium chloride solution (14 % NaCl) and the current density of 100 A/cm². High removal rates were obtained in similar chloride-nitrate electrolytes for VT9 and OT4-2 alloys [8]. At the same time, work [9] showed that the removal rate for titanium alloys was lower in the chloride-nitrate solutions as compared to the chloride solutions. According to [10], surface hydriding in electrolytes based on NH₄NO₃ is over the limit. According to other results of the study [11], hydriding of titanium alloys in chloride-nitrate
electrolytes is within tolerance. In comprehensive work [12], describing the extensive study of the machinability for titanium alloys, the prospects of chloride-nitrate electrolytes for machining compressor blades were verified in the context of the achieved high machining rate, as well as low surface layer roughness and hydriding values.

In article [13], the authors observed generation of the hydrided layer on the surface of the titanium alloy influenced by high temperature developed in the course of milling. Hydriding produced a considerable impact on the plastic properties of the alloy.

To sum up, a suggestion can be made that pure nitrates are of little use for titanium alloy ECM due to their high hydriding, but when combined with other components, they provide the enhancement of the technological characteristics. Among these electrolytes, sodium chloride and sodium nitrate (ammonium)-based electrolytes may be called as the most universal ones since the improved process performance as well as the reduced surface roughness and hydriding are observed in them. As opposed to multicomponent electrolytes, they comply with the operating requirements.

The issue of selecting a chloride-nitrate mixture for the pulse ECT has not been sufficiently studied until present regardless of its prospects. The results of the enhanced process performance study for these solutions are sparse and contradictory. The issue of the possibility to improve the precision, to reduce hydriding and to enhance surface roughness has not been addressed sufficiently.

3. Developing A Mathematical Model for The Hydriding Process in The Titanium Alloy ECM

The analysis was aimed at searching for the optimal electrolytes and pulse dimensional electrochemical machining conditions for the VT9 alloy as well as developing the theoretical surface hydriding model at the pulse electrochemical dimensional machining as well as development of the theoretical surface hydriding model in the pulse ECM.

The experimental studies of the surface hydriding during electrochemical machining were carried out on the experimental bench by employing cylindrical samples of the VT9 titanium alloy with the area of 100 mm². In the initial condition, the hydrogen content in samples was 0.005% and the surface roughness – Rₐ=2.5 µm. The following electrolytes were used for the analysis: sodium chloride, sodium nitrate, ammonium nitrate. The selection of electrolytes was limited by the most perspective electrolytes among the ones that were initially detected as ensuring the lowest gas emission, minimal hydriding, responding to the process selectivity requirements and ensuring low surface roughness with the pulse current ECM. It was shown that with a decrease of the gap down to 0.04–0.06 mm (within the range of ultra-small gaps, where the blade ECM bears the risk of short circuiting, and the increased process selectivity is possible at the same time) a tendency to the decreased hydriding is noted. That is, within the gap range where the ECT may really act as a finishing method, minimal hydriding may be achieved. Hydriding grows with the increased current pulse duration and pauses between the pulses. In the pulse ECM, the surface layer generation behaviour stipulating the hydriding during machining, will considerably differ from the direct current machining. It was established that the titanium alloy hydriding source in nitrate and chloride-nitrate solutions is hydrogen, which is generated in the anode. The hydriding mechanism was proposed based on the adsorption of anode atomic hydrogen and its penetration into the surface layer. The minimal contents of hydrogen in the surface layer may be obtained via the pulse ECM of titanium alloys in nitrate-based electrolytes. When using chloride-nitrate mixtures in optimal conditions, hydriding is ensured without exceeding the permissible limits. Adding superficially active materials (SAS) to electrolytes in the amount of 0.1...0.15 is useful for the pulse titanium alloy ECM. According to the analysis of the effect caused on the hydriding value by major ECM factors, it was defined that major factors are the duration and the rate of pulse, an electrode gap, total electrolyte concentration and electrode current. At the subsequent stage of the analysis, using the experiment planning, we determined the dependence of hydriding based on the above-mentioned factors. A fractional factorial experiment was performed with the use of the half-replicate 25-1 (16 experiments). The factor variation range is given in table 1.

Table 1. The range of conditions under study
Factors | Code factor symbol | Natural factor levels corresponding to the coded ones.
---|---|---
Pulse duration, ms, $t_i$ | $x_1$ | 0.06 | 2.0
Pulse rate, q | $x_2$ | 2 | 6
Gap a, mm | $x_3$ | 0.04 | 0.2
Electrolyte concentration, % | $x_4$ | 5 | 25
Voltage U, V | $x_5$ | 12 | 25

As a result of the above-mentioned analysis, the regression equation was obtained as follows:

$$y = 0.0527 + 0.00025X_1 + 0.000875X_2 + 0.00264X_3 + 0.00025X_4 - 0.00501X_5.$$ 

The equation adequacy as well as the significance of factors were checked. Using this dependence, it is possible, firstly, to determine the expected level of hydrogen content in the surface with the selected machining conditions in the context of the airfoil forming precision. Secondly, it allows adjusting the machining conditions towards lower hydriding values if they do not comply with the maximum permissible limit of hydrogen contents.

To obtain the universal generalized conclusions on the principal hydriding reduction directions, which would suit various alloy structures, conditions and electrolytes, we developed the mathematical model of the hydriding process in the titanium alloy ECM considering the joint effect caused by all the factors acting during machining and built with the adequate phenomenological model. In work [14], the authors studied the diffusion hydriding in conditions of the direct current ECM, and it was established that when increasing the surface dissolution rate, the density of the hydrogen diffusion flow directed inside the metal would grow and the quasi-stationary distribution of bound hydrogen would be set on the moving dissolution front. After the process termination, hydrogen accumulated in the surface layer continues diffusing into the depth of the metal, where it is transformed to the bound state.

For the pulse ECM, the large number of pulses $\sim 10^5–10^7$ equal in their intensity and supplied during the stock removal, are inherent. Small thickness of the layer dissolved within one pulse $h \sim 10^{-8}–10^{-4}$ mm, short pulses and pauses with the order of $\sim 10^{-4}–10^{-3}$ as compared to the characteristic relaxation times (the time for establishment of quasi-stationary moving front ($\sim 3–5$ s) and the time of hydrogen transition into the bound state ($\sim 60$ min)) allow considering this process as a sequence of momentary focused pulses.

Solving the task on determining the hydrogen concentration in a surface layer after the pulse ECM is reduced to determining the concentration balance on the moving dissolution surface. With the momentary removal of the layer with thickness $h$, the amount of hydrogen $N_p = h(C_h + C_p)$ is generated on a newly formed surface, where $C_h$ is the background hydrogen concentration in metal, $C_p$ is a reactive component stipulated by the anode dissolution electrochemical reaction. Furthermore, when the layer is dissolved, the excessive hydrogen in the amount of $N_e = \int C(z)dz$ contained in it is generated, where $\bar{C}(z)$ is the excessive hydrogen concentration established in the ECM. A fraction of this quantity of hydrogen equal to $\alpha(N_p + N_e)$, where $0<\alpha<1$, is the mass-transfer rate, enters the metal; the rest is transferred to the electrolyte solution, where a uniform concentration of reagents is maintained by mixing.

In turn, $\bar{C}(z)$ may be represented as a sum of free hydrogen $C'(z)$ and bound hydrogen (in the form of hydride) $C_\alpha(z)$: $\bar{C}(z) = C'(z) + C_\alpha(z)$. When considering a single flat pulse of power $\nu$ on
surface \( z=+0 \), the diffusion equation will take the form of:

\[
\frac{\partial C}{\partial t} = D \frac{\partial^2 C}{\partial z^2} (C - C_s) + \nu \delta(z,t), \tag{1}
\]

where \( \nu \) is a specific amount of hydrogen generated as a result of the layer dissolving of thickness \( h \), \( \delta(z,t) \) is the pulse function. The rate of free hydrogen flowing into the bound condition is determined by the first-order chemical reaction kinetics equation:

\[
\frac{\partial C_s}{\partial t} = A_x (C - C_s), \tag{2}
\]

where \( A_x \) is the averaged reaction constant for free hydrogen transformation into different bound conditions. If we pass on to \( C'(z) \) in equations (1) and (2), the result will be as follows:

\[
\frac{\partial C'}{\partial t} = D \frac{\partial^2 C'}{\partial z^2} - A_x C' + \nu \delta(z,t) \tag{3}
\]

\( z>0, t>0 \) with limiting condition \( \frac{\partial C'(z,t)}{\partial z} \bigg|_{z=0} = 0 \). \tag{4}

The equation of form (3) is reduced to the parabolic normal-form equation by substituting for \( C'(z,t) = u(z,t) \exp[-A_x t] \). Consequently, \( u(z,t) \) corresponds to solving the 2nd type heat conduction boundary problem for the half-line with pulse source \( \nu \) at the end:

\[
\nu \cdot \exp\left[ -\frac{z^2}{4 \cdot D \cdot t} \right]
\]

\[
\frac{\partial C'}{\partial t} = D \frac{\partial^2 C'}{\partial z^2} - A_x C' \tag{5}
\]

When multiplying (5) by \( \exp[-A_x t] \), the required free hydrogen concentration during the passage of one pulse will be obtained:

\[
C'(z,t) = G_i(z,t) = \frac{\nu \cdot \exp\left[ -\frac{z^2}{4 \cdot D \cdot t} - A_x \cdot t \right]}{\sqrt{\pi \cdot D \cdot t}}, \tag{6}
\]

where \( G_i \) is the function of the surface source - solution of equation (3) for the half-line. The concentration of bound hydrogen during the passage of one pulse will amount to:

\[
C_s(z) = A_x \int_0^z G_i(z,t) dt = \nu \sqrt{\frac{A_x}{D}} \exp\left[ -z \sqrt{\frac{A_x}{D}} \right]. \tag{7}
\]

During the passage of the following pulse, after time \( T \), a new surface hydrogen layer will be generated along with the disappearance of the previous one together with the dissolved layer with leaving traces:

\[
S' = G_i(z+h,t) = \frac{\nu \cdot \exp\left[ -\frac{(z+h)^2}{4 \cdot D \cdot t} - A_x \cdot t \right]}{\sqrt{\pi \cdot D \cdot t}}, \tag{8}
\]

\[
S_i = A_x \int_0^T G_i(z+h,t) dt =
\]

\[
= \frac{1}{2} \cdot \nu \sqrt{\frac{A_x}{D}} \left[ \exp\left[ -(z+h) \sqrt{\frac{A_x}{D}} \right] \cdot \text{erfc}\left[ \frac{z+h}{2 \sqrt{D \cdot T}} - \sqrt{A_x \cdot T} \right] - \exp\left[ -(z+h) \sqrt{\frac{A_x}{D}} \right] \cdot \text{erfc}\left[ \frac{z+h}{2 \sqrt{D \cdot T}} + \sqrt{A_x \cdot T} \right] \right], \tag{9}
\]
where \( \text{erfc}\xi = \frac{2}{\sqrt{\pi}} \int_\xi^\infty \exp\left(-x^2\right) dx \), \( S'(z) \) is the concentration of free hydrogen from the previous pulse at the moment of the subsequent one, \( S_x(z) \) is the concentration of bound hydrogen. \( S'_x(z) \) is the initial condition for the 2nd boundary value problem with the zero right side. Consequently, the concentration field of the momentary surface hydrogen source will take the form as follows:

\[
F_i(z, t) = \int_0^\infty S'(\xi, t) \frac{\exp\left[\frac{(z - \xi)^2}{4 \cdot D \cdot t} - A_\xi \cdot t\right] + \exp\left[\frac{(z + \xi)^2}{4 \cdot D \cdot t} - A_\xi \cdot t\right]}{2\sqrt{\pi \cdot D \cdot t}} d\xi,
\]

where \( F_i \) is the first-order phantom source. Then the concentration of bound hydrogen after two pulses as well as the full hydrogen concentration may be found:

\[
\overline{C}_i(z) = C_H + \alpha(C_p + C_H) - \frac{\sigma \exp\left[-z\sqrt{\frac{A_\xi}{D}}\right]}{\sqrt{1 + \sigma^2 - 2\alpha}},
\]

where \( \sigma = \frac{2A_\xi DT}{h} \). The total excessive amount of hydrogen:

\[
N(\sigma) = \int_0^\infty (C(z) - C_H) dz = \alpha \frac{A_\xi}{\sqrt{D}} (C_p - C_H) \frac{\sigma}{\sqrt{1 + \sigma^2 - 2\alpha}}.
\]

Function \( \phi(\sigma) = \frac{\sigma}{\sqrt{1 + \sigma^2 - 2\alpha}} \) has its maximum \( \sigma^* = \frac{\sqrt{1 - 4\alpha^2}}{2\alpha} \) in point \( \phi^* = \frac{1}{\sqrt{1 - 4\alpha^2}} \). From here, \( \alpha = \frac{1}{2\sqrt{1 + \sigma^2}} \). The maximum hydriding shall be expected at the dissolution rate of \( V_{dp} = \frac{h}{T} = \frac{4\alpha A_\xi D}{\sqrt{1 - 4\alpha^2}} \). So as to evaluate the hydrogen intensity, the total excessive quality of hydrogen in terms of the per unit area of the dissolution surface is used:

\[
N(\sigma) = \int_0^\infty (C(z) - C_H) dz = \alpha \frac{A_\xi}{D} (C_p - C_H) \frac{\sigma}{\sqrt{1 + \sigma^2 - 2\alpha}}.
\]

These characteristics allow determining the maximum performance of the technology and the direction of hydriding reduction as well as estimating mass-transfer rate value \( \alpha \). Thus, it follows from boundedness condition \( N \) at small \( \sigma \) that \( \alpha < 1/2 \). The developed model reflects the hydrogen surface saturation condition at the direct finishing completion moment, while, in practice, the described situation will always be distorted by the natural hydrogen diffusion inside the metal. The model indicates a way to achieving the minimal initial starting concentration of hydrogen during natural diffusion upon the ECM completion.

We have developed a method for determining the expected surface layer hydriding with the pulse ECM depending on the properties of the material being machined as well as machining conditions. It comprises the steps as follows:

1. A number of pulse ECM experiments are carried out for samples with the predetermined electrolyte in a wide range of machining conditions for the material being analysed in order to determine the metal dissolution rate; and the surface hydriding curves are defined. The evaluation of blade airfoil surface hydriding is proposed to be performed using the spectral method according to the procedure developed by the All-Russian Research Institute of Aviation Materials (VIAM).

2. A factor of \( \sigma \) and the value of total excessive amount of hydrogen \( N \) are determined for each experimental curve obtained.
3. Then, chart \( \phi = N(\sigma) \) is built. Function \( \phi(\sigma) = \frac{\sigma}{\sqrt{1 + \sigma^2 - 2\alpha}} \) has its maximum at point

\[ \sigma^* = \frac{\sqrt{1 - 4\alpha^2}}{2\alpha} \]

with the value of \( \phi^* = \frac{1}{\sqrt{1 - 4\alpha^2}} \), allowing us to experimentally determine the mass-transfer rate value: \( \alpha = \frac{1}{2\sqrt{1 + \sigma^2}} \). The value of kinetic constant \( A_k \) is determined as follows. By selecting random point \( z_0 \) on the experimental curve (figure 1) \( C(z) \), the following dependence shall be built:

\[ \psi(z) = \frac{C(z) - C_H}{C(z_0) - C_H}. \]  

(13).

![Figure 1. The dependence of hydriding along the depth of the surface layer on random point \( z^* \)](image)

When substituting (11) in (13), the following result is obtained: \( \psi(z) = \exp\left[\frac{A_k}{D}(z - z_0)\right] \). After taking logarithms of the last relation and making necessary transformations

\[ \frac{A_k}{D} = \frac{1}{z_0 - z} \ln \left( \frac{C(z) - C_H}{C(z_0) - C_H} \right), \]

from which

\[ A_k = D \left[ \frac{1}{z_0 - z} \ln \left( \frac{C(z) - C_H}{C(z_0) - C_H} \right) \right]^2, \]  

(14)

where \( A_k \) is the average value at experimental points of curve \( C(z) \). Value \( A_k \) is a constant for the material grade and its working type before the ECM, which has little dependence on the ECM mode with the absence of the intercrystalline etching to the depth of diffusion.

The last of remaining parameters \( C_p \) is determined by calculated values \( \sigma, \alpha, A_k, N(\sigma) \):

\[ C_p = \frac{N(\sigma) \cdot \sqrt{A_k \cdot (\sqrt{1 + \sigma^2 - 2\alpha})}}{\alpha \cdot \sqrt{D}} - C_H. \]  

(15)

The results of these studies and the hydriding mathematical model are planned to be used in developing a database for the EDM tool automated profiling module [15].

4. Conclusion

As a result of studies carried out, a dependence of BT-9 alloy ECM hydriding on the principal factors influencing the process (i.e. pulse duration, pulse rate, electrode gap, concentration of electrolyte and electrode current) was established. To define the universal dependence for the surface hydriding in...
titanium alloy ECM applicable to different structures of alloys, conditions and electrolytes, a mathematical model was developed for the surface layer saturation process in the pulse ECM. Based on the model, the method for determining the expected surface layer hydriding during pulse ECM depending on the properties of material being processed as well as machining conditions was developed. Also, the program for determining the expected surface layer hydriding in the pulse ECM depending on the properties of the material being machined as well machining conditions was developed and adjusted.

Acknowledgements
The authors of the article express their gratitude to the employees of engine-building companies in Samara region, the Russian Federation, for the assistance in the research.

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