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Abstract

The aim of investigations on the chapter was to determine regularities of solid solution hardening of surface layers of titanium alloys depending on the conditions of thermodiffusion saturation in rarified gas medium containing oxygen and determine the correlations between parameters of surface-hardened layers (surface hardness, depth of hardened zone, microstructure) and fatigue properties of titanium alloys under various methods of surface hardening. To achieve the formulated aim, the following methods were used: (a) thermodiffusion saturation of titanium alloys in rarified gas medium containing oxygen in the wide range of temperature-time and gas-dynamical parameters and (b) surface deformation by ultrasonic shock and shot-blasting treatments with rapid annealing of deformed surface by means of induction heating. The positive influence of surface hardening on the fatigue characteristics is decreased under the increasing of \( l \) when \( K \) is constant. The highest relative gain of fatigue strength \( (\Delta\sigma) \) of samples with CTT surface-hardened layers is marked for the low- and middle-strong alloys VT1-0 and OT4-1. Thus for alloy VT1-0, \( \Delta\sigma = 35\% \) under relative gain of surface hardness \( K = 70\% \) and \( l = 3 \, \mu m \). For the near-\( \alpha \)-alloy OT4-1, \( \Delta\sigma = 38\% \) under relative gain of surface hardness \( K = 35\% \) and \( l = 45 \, \mu m \)...

Keywords: titanium alloy, oxygen, mass gain, solid-solution hardening, fatigue strength

1. Introduction

An increasing of fatigue strength and endurance of titanium alloys of various structural classes can be achieved by the formation of surface layers with regulated structure and phase state during surface hardening processes of chemico-thermal treatment (CTT). CTT in the present time the thermodiffusion hardening of surface layers by interstitial impurities is not used to increase the fatigue properties of workpieces made of titanium alloys. However, works performed at PMI NASU have shown that the range of the parameter \( K \) exists where the endurance of alloys with gas-saturated layers is higher than fatigue endurance in the initial state. An optimal level of surface hardening depends on the phase-structural state of metal and relative depth of gas-saturated zone. The problem arises in the controlling of intensity of the physical and chemical processes in a “titanium alloy/gas medium” system to obtain the optimal phase-structural state of surface layers and increase the...
operating characteristics of metal. Therefore the aim of investigations on the second stage of the project was to determine (a) regularities of solid solution hardening of surface layers of titanium alloys (α, near-α, α + β) depending on the conditions of thermodiffusion saturation in rarified gas medium containing oxygen and (b) general regularities of influence of methods and regimes of surface deformation on phase, structural, and substructural state of various titanium alloys. It is expected that obtained results will allow to forecast the influence of the regime of CTT on the phase-structural state of surface layers of metal and level of hardening and determine the parameters of thermal treatments for achieving the regulated level of hardening.

Increasing of fatigue strength and durability of titanium alloy workpieces remains an actual modern problem. It is known that fatigue properties of titanium alloys can be increased sufficiently by means of optimization of phase-structural state of surface layer. The aim of the investigations of paper was to determine the correlations between parameters of surface-hardened layers (surface hardness, depth of hardened zone, microstructure) and fatigue properties of titanium alloys VT1-0, VT5, OT4-1, VT16, and VT22 under various methods of surface hardening: thermodiffusion saturation in gas medium containing oxygen (CTT). Determination of such correlations allow to define the parameters of surface hardening of titanium alloys necessary for increasing of fatigue properties and approximate these methods of surface hardening to the practical application.

2. Regularities of solid solution hardening of surface layers of titanium alloys (α, near-α, and α + β) depending on conditions of thermodiffusion saturation in rarefied gaseous medium containing oxygen

Accordingly, with the results obtained previously, the required level of solid solution hardening of surface layers of titanium alloys by interstitial impurities depends on the phase-structural state of metal and relative depth of hardened (gas-saturated) zone. In this connection the problem of purposeful control of intensity of physicochemical processes in the system “titanium (titanium alloy)/gas medium” for the formation of required phase-structural state of surface layers aimed to ensure the corresponding operating performance of materials (fatigue strength, endurance, etc.) arises. In turn, this envisages the study of kinetic regularities of interaction of titanium alloys in rarefied gas medium.

2.1 Kinetic parameters of interaction of VT1-0, VT5, OT4-1, and VT16 alloys with rarefied gas medium

As a result of interaction of titanium alloys with rarefied gas medium containing oxygen, the processes of sublimation and phase formation may take place [1]. The processes of gas saturation and phase formation will be predominated at selected temperatures (923...1023 K) and pressures (6.6 × 10⁻¹...6.6 × 10⁻² Pa), according to the analysis of changing of free energy of formation of solid solution of oxygen in titanium and titanium monoxide (Figure 1).

At the individual case, for alloys alloyed by manganese, molybdenum, and vanadium, the sublimation is possible due to the formation of oxide compounds with high pressure of saturated vapor [2]. Thus the gas saturation and phase formation increase the specimen mass of investigated titanium alloy, while sublimation decreases.
The influence of temperature-time and gas-dynamical parameters ($T = 650, 700, 750^\circ C$, $\tau = 1, 3, 5\, h$, $P = 6.6 \times 10^{-3}, 1.33 \times 10^{-2}, 6.6 \times 10^{-2}\, Pa$) of thermodiffusion saturation in controlled gas medium on the regularities of interaction of titanium alloys VT1-0, VT5, OT4-1, and VT16 is investigated by means of gravimetric analysis. The kinetic parameters of interaction of investigated titanium alloys determined by means of gravimetric analysis are shown in Tables 1–4.

According to the obtained results for $\alpha$-titanium alloys (VT1-0, VT5), the process of gas saturation is intensified with the increasing of interaction temperature and pressure of gas medium (partial pressure of oxygen). The $\alpha$-alloy VT1-0 (technical titanium) has the largest rate of interaction with rarefied gas medium containing oxygen under all conditions. Alloying of titanium by 5% Al-alloy VT5-slowed slightly the rate of mass gain under the same conditions of interaction.

The mass loss caused by intensification of sublimation of alloying element Mn is possible for near-$\alpha$-alloy OT4-1 (2% $\beta$-phase) under definite conditions of interaction with rarefied gas medium. The ratio of parameters $T$ and $P$ exists for this alloy when the rates of the gas saturation and sublimation processes become comparable. For the predomination of gas saturation processes, it is necessary to increase the partial pressure of oxygen or decrease the temperature of interaction.

Rate of gas saturation in rarefied gas medium decreased substantially with the increasing of quantity of $\beta$-phase in alloys (VT16 $\rightarrow$ VT22). This is caused by the decreasing of maximal solubility of oxygen in $\beta$-phase of titanium (6 at.%) in comparison with $\alpha$-phase (33 at.%). With the increasing of interaction temperature, this difference appeared most appreciably. Therefore, it was concluded that alloys with large quantity of $\beta$-phase are the ones less sensitive to the changing of the conditions of interaction with rarefied gas medium containing oxygen.

\begin{table} \centering
\begin{tabular}{|c|c|c|c|c|c|c|c|c|c|c|c|}
\hline
$T$, °C & \multicolumn{6}{c|}{$\Delta M/S$ (μg/cm$^2$) at residual pressure of gas medium} & \multicolumn{3}{c|}{\text{Residual pressure of gas medium}} \\
\hline
 & $P = 6.6 \times 10^{-3}\, Pa$ & $P = 1.33 \times 10^{-2}\, Pa$ & $P = 6.6 \times 10^{-2}\, Pa$ & $P = 6.6 \times 10^{-3}\, Pa$ & $P = 1.33 \times 10^{-2}\, Pa$ & $P = 6.6 \times 10^{-2}\, Pa$ \\
\hline
 & 1 h & 3 h & 5 h & 1 h & 3 h & 5 h & 1 h & 3 h & 5 h \\
\hline
650 & 3.46 & 9.905 & 15.98 & 4.58 & 12.9 & 20.61 & 7.95 & 21.34 & 33.14 \\
700 & 8.109 & 22.96 & 36.82 & 10.67 & 29.68 & 47.07 & 18.25 & 48.11 & 73.9 \\
750 & 17.416 & 48.83 & 77.85 & 22.82 & 62.68 & 98.64 & 38.45 & 99.49 & 151.23 \\
\hline
\end{tabular}
\caption{The specific mass gain of specimens of titanium alloy VT1-0 as a result of interaction with rarefied gas medium containing oxygen.}
\end{table}
Let us calculate the kinetic parameters as the function of temperature accordingly with the data of mass changing of specimens of titanium alloys in rarefied gas medium containing oxygen.

All kinetic dependences in the 5-h interval at the residual pressure \( P = 1.33 \times 10^{-2} \) Pa follow linear dependence (1) satisfactorily. This indicates that surface reactions at the “metal-gas” interface are the controlling stage of the processes [3]:

\[
\Delta M/S = (k^P \cdot \tau \pm A) \cdot 10^{-2}, \, [\text{g/m}^2]
\]

(1)

where \( k^P \) is the coefficient of linear rate under constant pressure and \( A \) is the confidence interval with a possibility of 0.98.

The coefficient of linear rate under isobaric conditions of thermally activated process depends on the absolute temperature \( T \) accordingly with Arrhenius equations [1]:

\begin{table}
\begin{tabular}{ccccccccc}
\hline
\( T, \, ^\circ\text{C} \) & \multicolumn{8}{c}{\( \Delta M/S (\mu\text{g/cm}^2) \) at residual pressure of gas medium} \\
& \( P = 6.6 \times 10^{-3} \) Pa & & & \( P = 1.33 \times 10^{-2} \) Pa & & & \( P = 6.6 \times 10^{-2} \) Pa & \\
& 1 h & 3 h & 5 h & 1 h & 3 h & 5 h & 1 h & 3 h & 5 h \\
\hline
650 & 2.24 & 6.53 & 10.65 & 2.57 & 7.44 & 12.10 & 3.31 & 9.48 & 15.31 \\
700 & 5.62 & 16.2 & 26.29 & 6.43 & 18.42 & 29.77 & 8.24 & 23.31 & 37.36 \\
750 & 12.83 & 36.64 & 59.07 & 14.64 & 41.49 & 66.60 & 18.68 & 52.14 & 82.86 \\
\hline
\end{tabular}
\end{table}

Table 2.
The specific mass gain of specimens of titanium alloy VT5 as a result of interaction with rarefied gas medium containing oxygen.

\begin{table}
\begin{tabular}{cccccc}
\hline
\( T, \, ^\circ\text{C} \) & \multicolumn{3}{c}{\( \Delta M/S (\mu\text{g/cm}^2) \) at residual pressure of gas medium} \\
& \( P = 6.6 \times 10^{-5} \) Pa & \( P = 1.33 \times 10^{-2} \) Pa & \( P = 6.6 \times 10^{-2} \) Pa \\
& 1 h & 3 h & 5 h \\
\hline
650 & -0.10 & -0.31 & -0.52 \\
700 & -0.16 & -0.49 & -0.82 \\
750 & -0.24 & -0.73 & -1.23 \\
\hline
\end{tabular}
\end{table}

Table 3.
The specific mass change of specimens of titanium alloy OT4-1 as a result of interaction with rarefied gas medium containing oxygen.

\begin{table}
\begin{tabular}{cccccc}
\hline
\( T, \, ^\circ\text{C} \) & \multicolumn{3}{c}{\( \Delta M/S (\mu\text{g/cm}^2) \) at residual pressure of gas medium} \\
& \( P = 6.6 \times 10^{-5} \) Pa & \( P = 1.33 \times 10^{-2} \) Pa & \( P = 6.6 \times 10^{-2} \) Pa \\
& 1 h & 3 h & 5 h \\
\hline
650 & 2.00 & 6.00 & 9.66 \\
700 & 2.32 & 7.00 & 11.60 \\
750 & 2.80 & 8.50 & 13.29 \\
\hline
\end{tabular}
\end{table}

Table 4.
The specific mass gain of specimens of titanium alloy VT16 as a result of interaction with rarefied gas medium containing oxygen.
\[ k^0(T) = B \cdot \exp\left(-\frac{E_{\text{екс}}}{RT}\right) \pm C, \quad \text{[g m}^{-2} \text{h}^{-1}] \] (2)

where \( B \) is the constant depending on temperature, \( E_{\text{екс}} \) is the total energy of process activation, and \( C \) is the confidence interval with a possibility of 0.98. Constants for Eqs. (1) and (2) and experimental activation energy of process are presented in Table 5.

All kinetic regularities follow linear dependence satisfactorily under isothermal conditions and various pressures accordingly with the data of thermogravimetry:

\[ \Delta M/S = (k^T \cdot \tau \pm F) \cdot 10^{-2}, \quad \text{[g/m}^2\text{]} \] (3)

where \( k^T \) is the coefficient of linear rate under constant temperature (700°C) and \( F \) is the confidence interval with a possibility of 0.98. Dependence of coefficient of linear rate under isothermal conditions on the residual pressure of medium is approximated satisfactorily by logarithmic dependence:

\[ k^T(P) = [H + J \cdot \ln(P)] \pm K, \quad \text{[g m}^2 \text{h}^{-1}] \] (4)

where \( H \) and \( J \) are the constants depending on pressure and \( K \) is the confidence interval with a possibility of 0.98.

Coefficients for Eqs. (3) and (4) are presented in Table 6.

### 2.2 Effect of temperature and time on surface metal hardness and hardening zone depth

Formation of interstitial solid solution in the metal during diffusion saturation of titanium alloys by gases in rarefied gas medium (mainly by oxygen) is bound up with strong distortion of crystallographic lattice (Figure 2) and as a result of this with essential increasing of hardness of metal. Therefore, the parameters of gas-saturated layers were determined by means of two methods: using microhardness

| Alloy | \( T, ^\circ \text{C} \) |
|-------|------------------|
|       | Using formula (1) | Using formula (2) |
|       | \( k^0, \text{g m}^{-2} \text{h}^{-1} \) | \( A, \text{g m}^{-2} \) | \( B, \text{g m}^{-2} \text{h}^{-1} \) | \( E_{\text{екс}}, \text{J mol}^{-1} \) | \( C, \text{g m}^{-2} \text{h}^{-1} \) |
| BT1-0 | 650 | 3.23 | 1.67 | 4 \times 10^7 | 7447.6 | 4.66 |
|       | 700 | 7.46 | 3.53 |
|       | 750 | 15.8 | 4.21 |
| BT5   | 650 | 2.44 | 0.59 | 1 \times 10^8 | 8140.6 | 3.91 |
|       | 700 | 6.02 | 2.96 |
|       | 750 | 13.49 | 4.10 |
| BT16  | 650 | 2.6 | 0.1 | 29.21 | 1350.8 | 1.07 |
|       | 700 | 2.86 | 0.87 |
|       | 750 | 3.3 | 1.10 |
| OT4-1 | 650 | 3.2 | 0.2 | 16585.2 | 4148.6 | 3.34 |
|       | 700 | 5.0 | 1.41 |
|       | 750 | 7.38 | 2.87 |

Table 5.
Kinetic parameters of gas saturation of titanium alloys under isothermal conditions.
that was measured on both surface and cross section of metallographic sample made of the gas-saturated specimen and changing parameters of crystallographic lattice. The last one method is more complex and laborious; therefore, it was used only for determination of parameters of maximally hardened layer. Depth of gas-saturated layer recognizes as a distance from the surface where increasing of hardness caused by dissolution of oxygen is equal to the measurement error.

The experimentally obtained results of influence of parameters of thermodiffusion saturation on the gain of surface hardness

\[ K = \left( \frac{H_s - H_b}{H_b} \right) \times 100\% \]

\( H_s \) is the surface hardness of metal and \( H_b \) is the bulk hardness) and depth of gas-saturated zone \( l \) determined by durometry.

The results concerning the determination of parameters of crystallographic lattice of specimens of investigated alloys after different regimes of thermodiffusion saturation are presented in Tables 15-18.

| Alloy | \( P \times 10^2 \) Pa | \( k \), \( g \cdot m^{-2} \cdot h^{-1} \) | \( F \), \( g \cdot m^{-2} \) | \( H \), \( g \cdot m^{-2} \cdot h^{-1} \) | \( J \), \( g \cdot m^{-2} \cdot h^{-1} \) | \( K \), \( g \cdot m^{-2} \cdot h^{-1} \) |
|-------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| BT1-0 | 0.66            | 7.46            | 1.25            | 24.357          | 3.3804          | 1.05            |
|       | 1.33            | 9.57            | 1.53            |                 |                 |                 |
|       | 6.6             | 15.2            | 2.24            |                 |                 |                 |
| BT5   | 0.66            | 5.3             | 0.64            | 10.243          | 0.98            | 0.77            |
|       | 1.33            | 6.02            | 0.90            |                 |                 |                 |
|       | 6.6             | 7.57            | 1.68            |                 |                 |                 |
| BT16  | 0.66            | 2.32            | 0.24            | 6.8862          | 0.9116          | 0.37            |
|       | 1.33            | 2.86            | 0.74            |                 |                 |                 |
|       | 6.6             | 4.41            | 1.05            |                 |                 |                 |
| OT4-1 | 0.66            | –0.16           | 0.063           | 25.189          | 4.8973          | 2.11            |
|       | 1.33            | 5.00            | 0.41            |                 |                 |                 |
|       | 6.6             | 11.56           | 0.77            |                 |                 |                 |

Table 6.
Kinetic parameters of gas saturation of titanium alloys under isothermal conditions.

Figure 2.
Changing of ratio of parameters of crystallographic lattice (a/c) of titanium alloy surface layer VT1-0 as a function of CTT regime: (1) in initial state, (2) after CTT: \( T = 650^\circ\text{C}, P = 6.6 \times 10^{-3} \text{ Pa}, \tau = 5 \text{ h} \), (3) after CTT: \( T = 750^\circ\text{C}, P = 6.6 \times 10^{-3} \text{ Pa}, \tau = 5 \text{ h} \).
The regularities of thermodiffusion saturation intrinsic for all investigated alloys were revealed basing on the analysis of obtained results, namely, parameters of gas-saturated layer $H_\mu$, $\Delta H_\mu$, and $l$ increase with the increasing of saturation time.

Table 7.
Gain of surface hardness of titanium alloy VT5 as a result of interaction with rarefied gas medium containing oxygen.

| T, °C | $K$ (%) at residual pressure of gas medium | $P = 6.6 \times 10^{-3}$ Pa | $P = 1.33 \times 10^{-2}$ Pa | $P = 6.6 \times 10^{-2}$ Pa |
|-------|---------------------------------|-----------------|-----------------|-----------------|
|       | 1 h    | 3 h    | 5 h    | 1 h    | 3 h    | 5 h    | 1 h    | 3 h    | 5 h    |
| 650   | 41     | 66     | 81     | 56     | 85     | 103    | 80     | 121    | 143    |
| 700   | 37     | 61     | 75     | 49     | 78     | 96     | 74     | 113    | 134    |
| 750   | 22     | 37     | 47     | 30     | 48     | 60     | 45     | 72     | 88     |

Table 8.
Gain of surface hardness of titanium alloy VT5 as a result of interaction with rarefied gas medium containing oxygen.

| T, °C | $K$ (%) at residual pressure of gas medium | $P = 6.6 \times 10^{-3}$ Pa | $P = 1.33 \times 10^{-2}$ Pa | $P = 6.6 \times 10^{-2}$ Pa |
|-------|---------------------------------|-----------------|-----------------|-----------------|
|       | 1 h    | 3 h    | 5 h    | 1 h    | 3 h    | 5 h    | 1 h    | 3 h    | 5 h    |
| 650   | 30     | 47     | 58     | 37     | 58     | 70     | 52     | 78     | 92     |
| 700   | 28     | 45     | 56     | 35     | 56     | 67     | 50     | 75     | 89     |
| 750   | 6.5    | 11     | 15     | 10     | 18     | 22     | 20     | 32     | 40     |

Table 9.
Gain of surface hardness of titanium alloy OT4-1 as a result of interaction with rarefied gas medium containing oxygen.

| T, °C | $K$ (%) at residual pressure of gas medium | $P = 6.6 \times 10^{-3}$ Pa | $P = 1.33 \times 10^{-2}$ Pa | $P = 6.6 \times 10^{-2}$ Pa |
|-------|---------------------------------|-----------------|-----------------|-----------------|
|       | 1 h    | 3 h    | 5 h    | 1 h    | 3 h    | 5 h    | 1 h    | 3 h    | 5 h    |
| 650   | 38     | 66     | 86     | 33     | 58     | 75     | 11     | 18     | 24     |
| 700   | 17     | 30     | 38.9   | 28     | 50     | 64     | 26     | 45     | 59     |
| 750   | 9      | 17     | 22     | 8      | 15     | 19     | 13     | 23     | 29     |

Table 10.
Gain of surface hardness of titanium alloy VT16 as a result of interaction with rarefied gas medium containing oxygen.

| T, °C | $K$ (%) at residual pressure of gas medium | $P = 6.6 \times 10^{-3}$ Pa | $P = 1.33 \times 10^{-2}$ Pa | $P = 6.6 \times 10^{-2}$ Pa |
|-------|---------------------------------|-----------------|-----------------|-----------------|
|       | 1 h    | 3 h    | 5 h    | 1 h    | 3 h    | 5 h    | 1 h    | 3 h    | 5 h    |
| 650   | 25     | 47     | 60.2   | 24     | 41     | 54     | 22     | 38     | 48.8   |
| 700   | 5      | 9      | 11.8   | 3      | 5      | 6.7    | 1      | 2      | 2.5    |
| 750   | 0.45   | 0.77   | 1      | 0.4    | 0.8    | 0.76   | 0.4    | 0.8    | 1      |

The regularities of thermodiffusion saturation intrinsic for all investigated alloys were revealed basing on the analysis of obtained results, namely, parameters of gas-saturated layer $H_\mu$, $\Delta H_\mu$, and $l$ increase with the increasing of saturation time.
under the same pressure of gas medium and temperature (Figures 3 and 4); the depth of gas-saturated zone \( l \) is increased, and value of relative gain of surface hardness \( \Delta H_{\mu}^{\%} \) is decreased with the increasing of saturated temperature in the range 650–750°C (Figure 5).
The first regularity is connected with the increasing of concentration of interstitial impurity in surface layer of metal with time and its penetration on the greater depth. The second regularity can be explained by acceleration of withdrawal of interstitial impurities from the surface due to the increasing of its diffusivity in $\alpha$- and $\beta$-titanium with the increasing of temperature (Figure 6). Under such conditions, the flow of oxygen from the medium to the metal surface becomes smaller in comparison with withdrawal flow owing to diffusion from the surface into the metal depth.

### Table 15.
Changing of parameter of crystallographic lattice of alloy VT1-0 as a result of interaction with rarefied gas medium containing oxygen.

| Operating mode of CTT | $a$  | $c$   | $c/a$ |
|-----------------------|------|-------|-------|
| Initial state         | 2.9481 | 4.6842 | 1.5889 |
| 75°C, $5.3 \times 10^{-4}$ Pa, 5 h | 2.9484 | 4.6860 | 1.5893 |
| 75°C, $1.3 \times 10^{-2}$ Pa, 5 h | 2.9496 | 4.6979 | 1.5927 |
| 75°C, $6.6 \times 10^{-2}$ Pa, 5 h | 2.948 | 4.7248 | 1.6027 |

### Table 16.
Changing of parameter of crystallographic lattice of alloy VT5 as a result of interaction with rarefied gas medium containing oxygen.

| Operating mode of CTT | $a$  | $c$   | $c/a$ |
|-----------------------|------|-------|-------|
| Initial state         | 2.9286 | 4.6746 | 1.5962 |
| 75°C, $6.6 \times 10^{-2}$ Pa, 5 h | 2.9281 | 4.6753 | 1.5967 |
| 75°C, $1.3 \times 10^{-2}$ Pa, 5 h | 2.9263 | 4.6729 | 1.5968 |
| 75°C, $6.6 \times 10^{-2}$ Pa, 5 h | 2.9329 | 4.7050 | 1.6042 |

### Table 17.
Changing of parameter of crystallographic lattice of alloy OT4-1 as a result of interaction with rarefied gas medium containing oxygen.

| Operating mode of CTT | $a$  | $c$   | $c/a$ | $Beta$ |
|-----------------------|------|-------|-------|--------|
| Initial state         | 2.9427 | 4.6823 | 1.5936 | 3.2265 |
| 75°C, $6.6 \times 10^{-2}$ Pa, 5 h | 2.9419 | 4.6856 | 1.5927 |        |
| 75°C, $1.3 \times 10^{-2}$ Pa, 5 h | 2.9426 | 4.6879 | 1.5931 |        |
| 75°C, $6.6 \times 10^{-2}$ Pa, 5 h | 2.9426 | 4.6911 | 1.5942 |        |

### Table 18.
Changing of parameter of crystallographic lattice of alloy VT16 as a result of interaction with rarefied gas medium containing oxygen.

| Operating mode of CTT | $a$  | $c$   | $c/a$ | $Beta$ |
|-----------------------|------|-------|-------|--------|
| Initial state         | 2.9287 | 4.6674 | 1.5936 | 3.2265 |
| 75°C, $6.6 \times 10^{-2}$ Pa, 5 h | 2.9298 | 4.6707 | 1.5942 | 3.2266 |
| 75°C, $1.3 \times 10^{-2}$ Pa, 5 h | 2.9284 | 4.6687 | 1.5942 | 3.2273 |
| 75°C, $6.6 \times 10^{-2}$ Pa, 5 h | 2.9301 | 4.6706 | 1.5940 | 3.2280 |
Figure 3.
Changing of the relative gain of surface hardness of titanium alloy VT1-0 under CTT ($T = 750^\circ C$, $P = 1.3 \times 10^{-3} \text{ Pa}$) depending on exposure time.

Figure 4.
Distribution of microhardness through the section of the specimens of alloy VT1-0 after CTT ($P = 1.3 \times 10^{-3} \text{ Pa}$, $\tau = 5 \text{ h}$) at temperatures (1) $650^\circ C$, (2) $700^\circ C$, (3) $750^\circ C$.

Figure 5.
Changing of the gain of surface hardness depending on interaction temperature with gas medium: (a) $P = 6.6 \times 10^{-3} \text{ Pa}$, (b) $P = 1.3 \times 10^{-2} \text{ Pa}$, (c) $P = 6.6 \times 10^{-2} \text{ Pa}$.
The phase composition of titanium alloys influences essentially on the quantitative index of saturation at not influencing on the qualitative appearance of determined regularities. The maximal dissolution of oxygen in α-phase of titanium composes 33 at.%, while in β-phase, only 6 at.%. Therefore in the titanium alloys with a large content of α-phase (VT1-0, VT5, and near-α-alloy OT4-1) during thermodiffusion saturation in gas medium, the gas-saturated layers with high gradient of hardness are formed (Figure 7).

Gas-saturated layer with high gradient of hardness is formed on the (α + β)-alloy VT16 at the low temperatures of saturation (Figure 8).

The values of coefficient $K$ representing the relative gain of surface hardness are higher sufficiently for α-alloys VT1-0 and VT5 in comparison with near-α-alloy OT4-1 and (α + β)-alloy VT16 under the same conditions of thermodiffusion saturation. On the other hand, the oxygen diffusion coefficient in β-titanium is high of order of magnitude in comparison with α-phase ($D_{\beta-Ti} = 3 \times 10^{-10}$ cm$^2$/s; $D_{\alpha-Ti} = 2 \times 10^{-9}$ cm$^2$/s at 800°C [3, 4]). Therefore, with the increasing in the alloys of volumetric content of β-phase, the depth of gas-saturated zone $l$ is increased. Thus on the surface of alloy VT16, the gas-saturated layers with a depth 2–2.5 times bigger than α-alloy VT1-0 are formed (160 and 60 μm correspondingly after treatment under condition $T = 750°C$, $P = 6.6 \times 10^{-2}$ Pa, $\tau = 5$ h).

Gas saturation and sublimation influence during thermodiffusion saturation not only the hardness but also the change of the state of surface and phase-structural state of near-surface layer of metal. Thus, because of sublimation and surface diffusion, the grain boundaries showed up; on some grains the characteristic step-like microrelief is developed (Figure 9).

**Figure 6.** Temperature dependence of oxygen diffusion coefficient in titanium [3].

**Figure 7.** Distribution of microhardness in the surface layer of alloy (a) VT5 and (b) OT4-1 after CTT ($T = 750°C$, $P = 6.6 \times 10^{-2}$ Pa, $\tau = 5$ h).
Gas saturation stabilizes the α-phase of titanium in the surface layer of metal and increases its hardness. The diffusion layer consists of α-phase rich layer and transition layer. The α-phase rich layer differs in structure from the base metal by increased content of α-phase that is easily revealed with metallography (Figure 10a). This layer is represented often by only one α-phase. Transition layer is not visibly different from the base metal (Figure 10b), but for this layer the larger hardness is inherent.

Saturation of surface layer by oxygen and in some cases the sublimation of alloying elements lead to the redistribution of alloying elements in the surface layer...
of metal and changing of its chemical composition on the alloy OT4-1, but on the other alloys, such changes are not observed (Figure 11).

2.3 Effect of oxygen partial pressure on the surface hardness and hardening zone depth

Apart from the temperature-time parameters, the gas-dynamical parameters of gas medium (partial pressure of chemically active components and dynamics—$I$ of leaking) influence the level of surface hardening. This influence should be taken into account during prediction of the consequences of thermodiffusion saturation of titanium alloys in the rarefied gas medium.

Let us consider in detail the influence of the changing of pressure of gas medium ($P = 6.6 \times 10^{-2}, 1.33 \times 10^{-2}, 6.6 \times 10^{-3} \text{ Pa, } I = 5 \times 10^{-3} \text{ Pa s}^{-1}$). The surface hardness and depth of hardened zone of most of titanium alloys are increased with the increasing of pressure under constant temperature accordingly with the obtained results (Tables 13 and 14 and Figure 12). This is most appreciably for $\alpha$-titanium alloys VT1-0 and VT5. The derivation from the regularity mentioned above is observed for near-$\alpha$-alloy OT4-1 and ($\alpha + \beta$)-alloy VT16 alloyed by elements with high volatility of oxides under saturation at 650°C (OT4-1, VT16) and 700°C (VT16)—decreasing of gain of surface hardness. In our opinion, this is caused by activation of sublimation process of Mn and V at relatively low intensity of gas saturation process and small solubility of oxygen in $\beta$-phase of titanium. At higher temperatures, the increasing of pressure of gas medium leads to the changing of tendencies in gain of surface hardness: the change in the inclination of temperature dependences is observed that can be connected with the increasing of oxygen flow from the medium, which becomes commensurable with the flow of withdrawal due to diffusion (Figure 12c).

Thermodiffusion saturation was performed under dynamic conditions of rarefied gas medium. That is to say, the residual pressure of medium is determined by dynamic equilibrium of gas flows pumped out and leaking into the reaction camera from the outside. The rate of leaking should be restricted because the increase of the
flow of the leaking gases influences the kinetics of interaction similarly to the increasing of pressure [5]. It should be noticed that all mentioned results are obtained under the conditions of low enough specific rate of leaking into the reaction chamber of vacuum equipment—\( I = 5 \times 10^{-5} \, \text{Pa s}^{-1} \). The increasing of leaking into the vacuum system intensifies sufficiently the oxidation and gas saturation of titanium alloys VT1-0, VT5, OT4-1, and VT16; however the regularities of this factor were not studied enough.

2.4 Relationship between the treatment parameters (pressure, temperature, duration) and level of interstitial solid

To predict the parameters of surface hardening of titanium alloys as a function of thermodiffusion saturation by interstitial impurities, the improved physicomathematical model of gas saturation of titanium alloys in rarefied gas medium is proposed for using [6].

Intensity of thermodiffusion processes is determined by phase-boundary reaction and concentration distribution of diffusant and diffusion coefficient. Phase-boundary reaction consists of a number of processes occurring in vacuum: adsorption of molecules and atoms of gases of residual atmosphere, their dissolution in the metal, oxidation, etc.; rate of this reaction is changed depending on the degree of medium discharging. Actually, the boundary concentration of oxygen on the surface of the metal is not being steadied at once but is being increased gradually with the rate depending on the degree of gas medium discharging. Based on the thermodynamic analysis (see first milestone), the titanium oxides are in the equilibrium with pressure of oxygen at all degrees of discharging that provides the boundary solubility of oxygen in \( \alpha \)-titanium, \( C_0 = 33 \text{ at.}\% \, \text{O}_2 \), that causes formation of stoichiometric oxides [3, 4]. Thus the calculation of diffusion saturation of metal by oxygen must be performed basing on the next boundary condition [6]:

\[
-D \frac{dC}{dx} = \alpha \cdot |C_0 - C(0, t)| \quad , \quad x = 0
\]  

(5)

where \( \alpha \) is the coefficient of rate of phase-boundary reaction, \( C_0 \) is the equilibrium concentration of oxygen in metal, \( C(0, t) \) is the actual concentration, and \( t \) is the time. These boundary conditions describe the mass change for period of time before the formation of oxide with certain thickness, which can already influence substantially the rate of the processes.

The function \( C(x, t) \) is the known solution of the diffusion task:
Mass changing of specimen under saturation by oxygen, dimension of diffusion zone up to 150 μm, and specimen thickness 3 mm per unit area is approximated by expression [5]:

\[
C(x,t) = C_0 \cdot \left[ \text{erfc} \left( \frac{x}{2 \sqrt{D} t} \right) - \exp \left( h x + h^2 D t \right) \cdot \text{erfc} \left( \frac{x}{2 \sqrt{D} t + h \sqrt{D} t} \right) \right] \text{, where } h = \alpha \cdot D
\] (6)

In the framework of physico-mathematical model of interaction of solid with gaseous medium under conditions of third type at the metal/gas interface, the coefficients of phase-boundary reaction (α) for alpha-titanium alloys VT1-0 and VT5 (see Tables 19 and 20) are determined using the experimental data (Tables 1 and 2).

The proposed approach for describing of the gas saturation processes with the use of coefficient of phase-boundary reaction α allows to calculate the mass change and concentration distribution of diffusant in the surface layer of metal under various temperature-time regimes of thermodiffusion saturation of α-titanium alloys, to determine the characteristics of gas-saturated layer (hardness distribution, depth of hardened zone) by using known correlation between hardness of surface layer and concentration of oxygen. The calculated nomograms for determination of permissible parameters of CIT of alloys VT1-0 and VT5 under condition of regulated level of surface hardening \( K = 25\% \) which is presented in Figure 13.

The obtained analytical data are in a good accordance with the experimental results that allow using this approach for evaluation and prediction of parameters of thermodiffusion hardening of surface layer of α-titanium alloys.

The additional investigation for determination of model dependences for near-α and (α + β)-titanium alloys is necessary since at the selected temperatures the change in the ratio of phase components in alloys and sublimation of alloying elements during gas saturation are possible.

| \( T, ^\circ C \) | \( \alpha \text{ (cm/s) at residual pressure of gas medium} \) |
|----------------|---------------------------------|
| | \( P = 6.6 \times 10^{-3} \text{ Pa} \) | \( P = 1.33 \times 10^{-2} \text{ Pa} \) | \( P = 6.6 \times 10^{-2} \text{ Pa} \) |
| 650 | \( 5.017 \times 10^{-9} \) | \( 5.792 \times 10^{-9} \) | \( 7.565 \times 10^{-9} \) |
| 700 | \( 1.206 \times 10^{-8} \) | \( 1.393 \times 10^{-8} \) | \( 1.819 \times 10^{-8} \) |
| 750 | \( 2.662 \times 10^{-8} \) | \( 3.073 \times 10^{-8} \) | \( 4.014 \times 10^{-8} \) |

Table 19. Coefficients of phase-boundary reaction (α) for titanium alloy VT1-0.

| \( T, ^\circ C \) | \( \alpha \text{ (cm/s) at residual pressure of gas medium} \) |
|----------------|---------------------------------|
| | \( P = 6.6 \times 10^{-3} \text{ Pa} \) | \( P = 1.33 \times 10^{-2} \text{ Pa} \) | \( P = 6.6 \times 10^{-2} \text{ Pa} \) |
| 650 | \( 1.76 \times 10^{-9} \) | \( 2.04 \times 10^{-9} \) | \( 2.66 \times 10^{-9} \) |
| 700 | \( 4.48 \times 10^{-9} \) | \( 5.18 \times 10^{-9} \) | \( 6.76 \times 10^{-9} \) |
| 750 | \( 1.04 \times 10^{-8} \) | \( 1.20 \times 10^{-8} \) | \( 1.56 \times 10^{-8} \) |

Table 20. Coefficients of phase-boundary reaction (α) for titanium alloy VT5.
3. Correlation between parameters of surface strengthening layers of different titanium alloys and their fatigue properties

3.1 Correlation between parameters of surface strengthening (surface hardness, hardening zone depth) and the fatigue properties of different titanium alloys under CTT

To determine the correlation between parameters of surface hardening and fatigue properties of titanium alloys of various structural classes under conditions of thermodiffusion saturation in controlled gas medium in the surface layers of metal, (a) the hardened layers of different depth with identical level of surface hardening, $K = \text{const}$, $l = \text{var.}$, and (b) the hardened layers of identical depth with different level of surface hardening, $K = \text{var.}$, $l = \text{const}$, were formed. It allows to reveal the influence of level and depth of surface hardening and also determine the optimal combination of these parameters.

3.1.1 $\alpha$-Alloy VT1-0

3.1.1.1 Regulated surface hardening of VT1-0 titanium alloy under conditions of thermodiffusion saturation in controlled gaseous medium

To form the hardened layers with various ratios $K$ and $l$, the determined relations considering the influence of temperature-time and gas-dynamical parameters of rarefied gas medium containing oxygen on the parameters of hardened layers and peculiarities of solid solution surface hardening of titanium alloys of various structural classes were used [7–12]. The used approaches for selection of parameters of thermodiffusion saturation under specified $K$ and $l$ are demonstrated in Figure 14 for alloy VT1-0 as an example.

Thus, the hardened layers of different depths with identical level of surface hardening can be obtained by means of changing the oxygen partial pressure and duration of saturation. The change of temperature influences the intensity of processes of thermodiffusion saturation. The influence of rate of oxygen leaking into the reaction chamber corresponds to the changing of partial pressure of chemically active component of gas medium (Figure 15). In addition the peculiarities of thermodiffusion saturation of titanium alloys of various structural classes should be taken into account. Thus, the hardened (gas saturated) layers with predetermined ratios $K$ and $l$ can be obtained due to changing of four parameters of thermodiffusion saturation ($T$, $\tau$, $P_{O2}$, and $I_l$—rate of oxygen in leakage into the reaction camera).
The parameters of thermodiffusion saturation regimes of titanium alloy VT1-0 in controlled gas medium containing oxygen which are selected based on the approach mentioned above which are refined experimentally and correspond to normative documents [13] are presented in Table 21. Parameters of surface-hardened layer and hardness distribution through the cross section of samples are determined by means of durometry.

3.1.1.2 Influence of CTT on fatigue properties of \(\alpha\)-titanium alloy VT1-0

The parameters of thermodiffusion saturation regimes of titanium alloy VT1-0 in controlled gas medium containing oxygen which are selected based on the approach mentioned above which are refined experimentally and correspond to normative documents [13] are presented in Table 21. Parameters of surface-hardened layer and hardness distribution through the cross section of samples are determined by means of durometry.

The surface-hardened layers influence sufficiently the metal fatigue resistance. The results of fatigue tests of samples of alloy VT1-0 after regulated surface hardening by thermodiffusion saturation in gas medium are presented in Figures 16–19 and Tables 22 and 23. The obtained results allow analyzing the influence of the level of surface hardening and depth of hardened zone on the metal fatigue resistance.

![Figure 14](image-url) Example of a choice of VT1-0 titanium alloy thermodiffusion saturation’s parameters: (1) \(P = 6.6 \times 10^{-4} \text{ Pa}\), (2) \(P = 1.33 \times 10^{-2} \text{ Pa}\), and (3) \(P = 6.6 \times 10^{-3} \text{ Pa}\).

![Figure 15](image-url) Influence of inleakage rate \((I_{\text{in}})\) on VT1-0 titanium alloy surface hardness gain.
3.1.1.3 Influence of level of surface hardening

Fatigue strength $\sigma_{fr}$ of titanium alloy VT1-0 is being increased initially (Table 2 and Figure 17) and then decreased with the increasing of level of surface hardening $K$ from 5 to 90%, at constant depth of hardened zone ($l = 30–35 \, \mu m$). In other
Figure 16. Fatigue curves of titanium alloy VT1-0, under rotating bending conditions, depending on the level of surface hardening when depth of hardened zone (gas saturated) is constant: (1) initial state $K = 5\%; l = 5\, \mu m$; (2) $K = 25\%; l = 30\, \mu m$; (3) $K = 50\%; l = 30\, \mu m$; (4) $K = 70\%; l = 30\, \mu m$; (5) $K = 90\%; l = 30\, \mu m$.

Figure 17. Fatigue strength of titanium alloy VT1-0, under rotating bending conditions, as a function of level of surface hardening when depth of hardened zone (gas saturated) is constant $l = 30\, \mu m$.

Figure 18. Fatigue curves of titanium alloy VT1-0, under rotating bending conditions, depending on depth of hardened (gas saturated) zone, when level of surface hardening is constant (a, $K = 50\%$; b, $K = 70\%$; (4) initial state $K = 5\%; l = 5\, \mu m$, (2) $l = 30\, \mu m$, (3) $l = 70\, \mu m$. 

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words, fatigue strength has a maximum. Relative gain of fatigue strength $\Delta \sigma_{-1}$ is the highest when $K = 70\%$. Such character of changing $\sigma_{-1}$ can be explained by the improvement of fatigue properties due to dissolution of oxygen in metal with formation of solid solution that is accompanied by the appearing of compressing stress. On the other hand, metal is embrittled due to solid solution hardening by oxygen dissolution. One or another factor dominates the defined conditions,

$$\Delta \sigma_{-1}/C_{0}$$ is the highest when $K = 70\%$. Such character of changing $\sigma_{-1}/C_{0}$ can be explained by the improvement of fatigue properties due to dissolution of oxygen in metal with formation of solid solution that is accompanied by the appearing of compressing stress. On the other hand, metal is embrittled due to solid solution hardening by oxygen dissolution. One or another factor dominates the defined conditions,
therefore increasing of fatigue properties is possible up to a certain level of hardening higher of which fatigue properties is being decreased.

3.1.1.4 Influence of depth of hardened zone

Relative gain of fatigue strength is being decreased with increasing of depth of hardened zone $I$ under constant level of surface hardening $K$ (Figures 18 and 19, Table 3). The higher level of $K$, the rather fatigue strength of alloy is being decreased with the rising of depth of hardened zone (Figure 19).

Solid solution hardening of surface layer of titanium alloys under conditions of thermodiffusion saturation by interstitial impurity (oxygen) can lead to the embitterment of hardened in this way layer and its brittle failure under conditions of the repeated loading. Because of this the investigations of fracture of samples were carried out after fatigue tests by rotating bending. Results of fractographical investigations of near-surface parts of fractures of titanium alloy VT1-0 samples with different levels of surface hardening after fatigue tests by rotating bending are presented in Figure 20.
Cup-shaped fracture mode is fixed on the fractograms of near-surface layer of samples that testify in ductile failure (Figure 20a–c). That is, the total embrittlement of surface layer does not take place even if the level of fatigue strength is maximal (Figure 7a). Brittle mode of fracture of thin (~1...2 μm) near-surface layer is observed only on the specimen with $K = 70\%$, $l = 70$ μm (Figure 20d and e). The time till origin of fatigue crack under alternate stress is being decreased providing that such layer exists on the samples and in turn the fatigue strength is being decreased. The decreasing of fatigue strength of alloy VT1-0 hardened by CTT with the increasing of depth of hardened zone $l$ at a high level of surface hardening $K$ is connected with this fact.

Thus, it can be concluded that for each of surface hardening level $K$ of titanium alloy VT1-0 under conditions of thermodiffusion saturation in controlled gas medium, an optimal depth of hardened (gas saturated) zone $l$ ensuring the highest level of fatigue characteristics exists. And vice versa for each depth of hardened zone, the optimal level of surface hardening exists. The aim of the next work stage is to search the optimal ratio of parameters $K$ and $l$.

### 3.1.2 α-Alloy VT5

The parameters of regimes of thermodiffusion saturation of titanium alloy VT5 in the controlled gas medium containing oxygen and parameters of surface-hardened layer and cross section hardness distribution are presented in Table 24.

#### 3.1.2.1 Influence of CTT on the fatigue properties of α-alloy VT5

The results of fatigue tests of samples of alloy VT5 after regulated surface hardening by thermodiffusion saturation in the gas medium are presented in Figures 21–23 and Table 25.

It should be noticed that in the presented case, the character of dependence of fatigue strength $\sigma_{-1}$ on the level of surface hardening $K$ has a maximum level which depends on the depth of hardened zone $l$ (Figure 22). The relative gain of fatigue strength $\Delta \sigma_{-1}$ of alloy VT5 reaches 20% under conditions $K = 19\%$, $l = 45–50$ μm.

The fatigue strength is being decreased with the increasing of depth of gas-saturated layer under constant $K$ (Figure 23).

It should be a supposition that under the analyzing of dependences presented in Figures 22 and 23, the maximal gain of fatigue strength $\Delta \sigma_{-1}$ for alloy VT5 can be reached by the creation of gas-saturated layer of parameters $K \approx 45–55\%$, $l \approx 40–50$ μm. Such parameters of gas-saturated layer can be determined as optimal parameters of the hardening of alloy VT5. It is the determination of optimal parameters of hardening to provide the highest gain of fatigue strength that is the aim of the next project step.

### 3.1.3 Near-α-alloy OT4-1 and (α + β)-alloy VT16

The regularities of the analogue presented above (see Tables 26 and 27) are observed for the near α-alloy OT4-1 and (α + β)-alloy VT16. The sufficient effect of solid solution hardening is observed for alloy OT4-1: relative fatigue strength gain $\Delta \sigma_{-1}$ reaches 38% under relative gain of surface hardening $K = 35\%$, $l = 45–50$ μm. It can be concluded that for titanium alloys with low or middle level of strength (VT1-0 and OT4-1 alloys), the positive effect of thermodiffusion saturation of metal surface layers by interstitial impurities is the highest: $\Delta \sigma_{-1} = 35–40\%$.
The sensitivity of titanium alloys to the presence of gas-saturated layers is increased with the increasing of $\beta$-phase value. As a result the increasing of fatigue characteristics is attained under smaller values of relative gain $K\%$. The value of fatigue strength gain is being decreased. Results of VT16 alloy tests can be a confirmation (Table 27).

Thus it can be concluded that for each level of surface hardening $K$ of investigated titanium alloys VT1-0, VT5, OT4-1, and VT16 under conditions of thermodiffusion saturation in controlled gas medium, the optimal depth $l$ of hardened (gas-saturated) zone exists which provides the highest level of fatigue
characteristics. In addition, vice versa for each depth of hardened zone, the optimal level of surface hardening exists. Furthermore, it can be forecasted that for each alloy the optimal relation between parameters $K$ and $l$ exists that provides absolutely maximal gain of fatigue strength. Such parameters of gas-saturated layers can be determined as optimal parameters of alloy hardening. The aim of the next step lies in the search of the optimal level of $K$ and $l$ parameters of the hardened zone.
4. Conclusions

1. The kinetic parameters of interaction and regularities of solid solution hardening of titanium alloys VT1-0, VT5, OT4-1, and VT16 under conditions of thermodiffusion saturation in rarefied gas medium are determined.

2. It is shown that under the same conditions of saturation ($T$, $\tau$, $P$), the hardened layers of various parameters ($H$, $I$) are formed on the titanium alloys. The monophase $\alpha$-titanium alloys VT1-0 and VT5 and near-$\alpha$-alloy OT4-1 are the most sensitive to the conditions of gas saturation: the gain of surface hardness and its gradient in the hardened layer increase sufficiently. With the increasing of $\beta$-phase (OT4 $\rightarrow$ VT16), changing of the parameters of CTT has less
influence on the hardness of surface layer, but the depth of the hardened zone is being increased with the increasing of the temperature and exposure time.

3. It is determined that solid solution hardening of titanium alloys VT1-0, VT5, OT4-1, and VT16 under conditions of thermodiffusion saturation by interstitial impurities in controlled gas medium leads to the increasing of fatigue strength under a definite ratio of hardened layers $K$ and $l$. The character of dependence of fatigue strength ($\sigma_{-1}$) of investigated alloys on the level of surface hardening ($K$) has a maximum value which depends on the depth of hardened zone ($l$). The positive influence of surface hardening on the fatigue characteristics is decreased under increasing of $l$ when $K$ is constant. The highest relative gain of fatigue strength ($\Delta\sigma_{-1}$) of samples with ChTT surface-hardened layers is marked for the low- and middle-strong alloys VT1-0 and OT4-1. Thus for alloy VT1-0, $\Delta\sigma_{-1} = 35\%$ under relative gain of surface hardness $K = 70\%$ and $l = 30 \mu m$. For the near-$\alpha$ alloy OT4-1, $\Delta\sigma_{-1} = 38\%$ under relative gain of surface hardness $K = 35\%$ and $l = 45$–$50 \mu m$.

4. The surface hardening of titanium alloy VT1-0 by thermodiffusion saturation (within the limits of parameters $K$ and $l$) provides saving of enough reserve of plasticity and does not influence the character of metal failure accordingly with the fractographical investigations. The failure of surface-hardened layer takes place by ductile plastic mechanism accordingly with fractographical investigations.

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