Mechanism of Self-Ignition
of Titanium Alloys in Oxygen

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It is proposed that the ignitability of titanium alloys in oxygen, characterized by critical ignition pressure, is determined by critical temperature, i.e., the temperature to which juvenile surface fragments can be heated during metal fracturing. This hypothesis is supported by comparison of calculated critical temperatures with critical ignition pressures measured experimentally for various titanium α-alloys.
Key words: titanium alloys, juvenile surface, oxygen, self-ignition, fracture.

Titanium and its alloys with their high specific strength and corrosion properties are the most promising materials for constructing reactors for the chemical industry and nonferrous metallurgy. However, wide technological applications of these materials is hindered by the potential risk of failure of titanium equipment due to the abnormal ignitability of titanium and its alloys in oxygen and oxygen mixtures. Self-ignition occurs only in the case of fracture (even local) of construction metal and contact of a juvenile metal surface (newly formed, free from an oxide film) with oxygen media at pressures higher than a certain critical pressure p^*. Since such fracture (for example, because of friction or scratching) cannot be ruled out during operation of reactors, especially those with moving parts, equipment made of titanium and its alloys can be safely operated in an oxygen medium only if the process parameters exclude the possibility of self-ignition of the materials with juvenile surfaces.

The conditions of self-ignition of titanium alloys in oxygen and oxygen mixtures have been the subject of a number of experimental studies [1–10], which established the following:

— Ignition is possible for various types of fracture, such as tension [1–9], flexure [5], impact [4, 9] as a result of friction [4], rupture by oxygen pressure [5], an electric spark discharge [10], and exposure of a specimen to a high-rate oxygen flow [7–9]. The critical ignition

pressure $(p^*)^2$ of an alloy is different for each type of fracture;

— Ignition occurs only as a result of direct metal fracture, whereas elastic and plastic deformations under stresses smaller than fracture stress do not lead to ignition of a new juvenile surface [2, 5];

— Practically all commercial titanium alloys tend to ignite [2, 3, 9]; the critical pressures of ignition in oxygen for the same fracture type differ by more than an order of magnitude and vary from 0.7 MPa for OT4-2 alloy of the Ti–Al–Mn system to 7.5 MPa for titanium iodide (99.98% Ti) [2, 3];

— The values of p^* for alloys depend on the character and relief of the fractured surface; the critical pressure of alloy ignition in oxygen increases during transition from tough fracture (uneven fracture) to brittle fracture (flat fracture). For this reason, preliminary hydrogenation or nitration of titanium alloys [2, 3] and incision of a specimen (to get a stress concentrator), which promotes transition to brittle fracture, lead to an increase in critical ignition pressure: for example, for VT1-0 alloy, the pressure p^* increases from 2.3 to 5.0 MPa in transition from rupture of intact specimens to specimens with incisions (author's data);

— Ignition occurs in both liquid and gaseous oxygen in the temperature range of 90 K [4] to 1273 K [2]. As the experimental temperature T_0 increases, the values of p^* of alloys usually decrease [1–5] but, sometimes,

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[2]p^* is the minimal oxygen pressure or partial oxygen pressure in a mixture with an inert gas at which the appearance of a juvenile surface leads to self-ignition.

can also grow [5, 9]. The geometrical dimensions of examined constructions have a contradictory effect on p^*: as the cross-sectional area of ruptured specimens increases, the pressure p^* of alloys can remain constant [1], decrease, or increase [5];

— Dilution of gaseous oxygen with an inert gas or water vapor always leads to an increase in critical ignition pressure [1–5].

However, despite abundant experimental data, the theoretical basis for self-ignition of titanium in oxygen has been developed insufficiently, which hinders prediction of safe use of titanium materials in industry and reliable prediction of the self-ignition ability for new alloys.

Littman [1] suggested that metallic materials (titanium, zirconium, and their alloys) are ignitable in oxygen only if their oxides are soluble in liquid metals. According to this hypothesis, self-ignition occurs only if the initial reaction is intense enough to raise the surface temperature to the melting point for this metal. According to Borisova and Bardanov [2], contact of a pure (unoxidized) titanium surface with oxygen causes exothermic oxidation involving the formation of an oxide layer and release of a large amount of heat. At low oxidation rate, for example, in air, the heat of the reaction disperses into the ambient medium. At higher oxidation rates (in pressurized oxygen), the heat of the reaction is accumulated and the metal is ignited. It is believed [10] that the main reason for titanium ignition in oxygen is damage of an oxide film and free access of oxygen to the metal surface. Since the rate of titanium oxidation depends on oxygen pressure and the temperature in the system, a certain oxygen temperature corresponds to a certain oxygen pressure at which the critical oxidation rate is so high that the oxidation yields interaction products that have a random structure and do not prevent further oxygen access to the juvenile surface.

However, explaining the possibility of self-ignition of materials by the solubility of their oxides in liquid metals, the hypothesis of [1] does not explain why juvenile surfaces of titanium, zirconium, and their alloys, unlike other metals, enter an exothermic reaction with oxygen whose rate is sufficient to heat the surface to the metal melting point even at room temperature. Yet, as was shown by later studies [11, 12], other metallic materials, whose oxides are virtually insoluble in liquid metals, such as chromium–nickel alloys, steels, and iron, can ignite after fracture in oxygen. On the other hand, the explanations of [2, 10] are based on the fact that interaction of a free titanium surface with oxygen leads to the formation of an oxide layer at the ignition moment, i.e., this interaction pattern is close to oxidation whose

rate, as the authors believe, depends on oxygen pressure. At the same time, it is known [13, 14] that the rate of titanium oxidation over a wide temperature range is determined by the rate of oxygen diffusion through the TiO_2 layer and does not virtually depend on pressure (at least, at $p_{O_2} \geq 45$ Pa [13]). However, self-ignition of fractured titanium constructions cannot be explained only by chemical interaction of a new juvenile surface with oxygen because it does not explain the above experimental phenomena, in particular, different ignition ability (p^*) of titanium alloys. For example, it remains unclear why the critical ignition pressures of materials similar in composition and oxidation rate, such as titanium iodide (99.98% Ti) and commercial titanium (99.0% Ti), differ by more than a factor of three [2].

In the present paper, using literature data, results of our experiments, and an analysis of physicomechanical energy conversions, we propose the hypothesis on the self-ignition of constructions of titanium and its alloys during fracture in oxygen. This hypothesis is based on the following data:

— At room temperature (especially at cryogenic temperature) at which titanium constructions ignite, the rate of interaction of titanium and its alloys with oxygen is low; it increases only at temperatures above ≈ 623 K [13, 14];

— Self-ignition, at least at room temperature, is possible only if the metal is fractured; if the oxide film is damaged (for example, scratched), titanium does not ignite;

— During high-rate fracturing, metals are heated. It is well known that metal chips can heat to several hundred degrees during turning of metal constructions. In our experiments, titanium specimens strained in an inert medium were heated to $\Delta T = 70$ K, as was recorded by thermocouples placed near the fracture site on the metal surface. Close temperatures ($\Delta T = 55$ K) are reached for an aluminum alloy strained in liquid helium [15];

— Microscopic observations of specimens fractured in oxygen show that fracture sites at which heat transfer to the depth of the specimen is obstructed are commonly the first to ignite (completely or partly separated particles, burrs, micropeaks, etc.).

The aforesaid led us to the conclusion that only those elements of a juvenile surface tend to ignite that have been heated to a high temperature T^* by the moment of interaction with oxygen. The temperature T^* is the sum of the initial temperature T_0 and the temperature increment ΔT due to heat release during metal

fracturing[3]: $T^* = T_0 + \Delta T$. In this case, the temperature T^* to which elements of a new juvenile surface are heated is a critical parameter that characterizes the ability of a titanium alloy to ignite. At equal initial temperatures ($T_0 = $ const), this parameter is the temperature increment ΔT due to the heat release during metal fracturing. As our experiments show, since the ignition ability of titanium alloys depends uniquely on oxygen pressure, the critical ignition pressure of the alloys is likely to depend on the critical ignition temperature T^* to which elements of the juvenile surface can be heated upon fracture.

HEAT ESTIMATES

We considered the possible heating ΔT and the critical temperature T^* of fractured elements of various titanium alloys strained to rupture in comparison with experimental values of p^* for the alloys in oxygen.

The value of ΔT was calculated using calorimetric measurements of [17] for low-carbon steel, which show that during plastic deformation of cylindrical rods of tough metals, more than 90% of the work transforms to heat. The work done by plastic deformation (A_{fr}) by the rupture moment was calculated as the product of the fracture load P_{fr} and the path dL along which the work was performed. If the fracture volume is found as $V = FdL$ (F is the specimen cross section at the rupture site), the specific fracture work was found as $A_{fr}/V = P_{fr}dL/FdL = P_{fr}/F = S_k$, where S_k is the true fracture stress (true fracture toughness).

Since the values of S_k are not known for all materials, this characteristic was calculated in accordance with [18] using the conventional ultimate strength (σ_{ign}) and the relative narrowing of the cross-sectional area (ψ) for specimens extended with considerable concentrated strain:

$$S_k = \sigma_{ign}(0.8 + 2.06\psi). \qquad (1)$$

We assumed that at the moment of fracture, the specimen's neck fails almost instantaneously and the entire fracture work (A_{fr}) transforms to heat, expended on heating the fractured metal volume. In this case, for some elements with obstructed heat transfer, this heating is almost adiabatic and is calculated by the formula $\Delta T \cong A_{fr}k/V\rho\bar{c}_p$. After substitution of the specific work, this formula becomes

$$\Delta T = S_k k/\rho\bar{c}_p, \qquad (2)$$

where k is a coefficient that characterizes the fraction of the work A_{fr} transformed to heat (if $k \cong 1$), ρ is the metal density, and $\bar{c}_p$ is the average heat capacity of the metal in the analyzed temperature range.

Table 1 shows calculated S_k, ΔT, and T^* for various titanium alloys. It can be seen that individual fracture elements of all alloys can be heated to high temperatures ($\Delta T \leq 470$ K) by the heat released during plastic strain of the metal.

Below, we give a possible explanation of the effect of various factors on p^* from the viewpoint of the above hypothesis that the quantity p^* depends on the temperature level T^* to which elements of the juvenile surface formed can be heated during fracture.

EFFECT OF COMPOSITION AND PHYSICOMECHANICAL PROPERTIES OF ALLOYS ON THE CRITICAL IGNITION PRESSURE

The calculation results were compared with refined results of experiments on ignition of various titanium α-alloys reported in [9].[4] Specimens of these materials were extended with considerable concentrated strain and had fractured surfaces typical of tough fracture (see Table 1 and Fig. 1).

A comparison of the data of Table 1 and the curve in Fig. 1 shows that with increase in the final temperature T^* (from 682 to 770 K) to which juvenile surface fragments can be heated, the critical oxygen pressure required to ignite the specimens decreases monotonically (from 2.9 to 0.7 MPa). At constant experimental temperature ($T_0 = 293$ K), the heating of the specimens correlates similarly to the critical pressure: as ΔT increases from 405 to 470 K, the ignition pressure decreases from 2.3 to 0.9 MPa. Since ΔT depends on the fracture work, which, in turn, depends on the fracture stress (2), the results of Table 1 indicate the dependence of the critical ignition pressure on the strength characteristics of the material: the higher the stress necessary for fracture of the specimen, the lower the oxygen pressure at which the metal juvenile surface is ignited.

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[3]This assumption is in agreement with the hypothesis of [16] that the adiabatic heating accompanying high-rate fracture of a construction may be responsible for self-ignition of titanium in oxygen.

[4]In [9], cylindrical rods with diameter $d_0 = 3$ mm made of various alloys were uniaxially strained in oxygen under various pressures at experimental temperatures $T_0 = 293$ and 473 K. For specimens of each alloy that were not ignited, the relative narrowing of the cross-sectional area was calculated by measuring the diameter before (d_0) and after (d_{fr}) the rupture: $\psi = (d_0^2 - d_{fr}^2)/d_0^2$. The arithmetic mean of this quantity was then used in the calculations.

TABLE 1

Calculated Values of S_k, ΔT, and T^* for Titanium α-Alloys
at Various Test Temperatures (T_0) in Comparison with Experimental
Values of the Critical Ignition Pressure in Oxygen (p^*)

Alloy	T_0, K	p^*, MPa	σ_{ign}, MPa	ψ	S_k, MPa	ΔT, K	T^*, K
VT1-0	473	2.9	250	0.73	576	209	682
VT1-0	293	2.3	470	0.73	1083 (966*)	405	698
Ti–15Zr	293	1.6	540	0.65	1155	449	742
OT4-1	293	1.5	680	0.44	1160	442	735
PT17	293	1.1	925	0.26	1235	455	748
PT3V	293	0.9	770	0.41	1266	470	763
OT4-1	473	0.7	450	0.56	879	297	770

Note. The data of [19] are marked by asterisk.

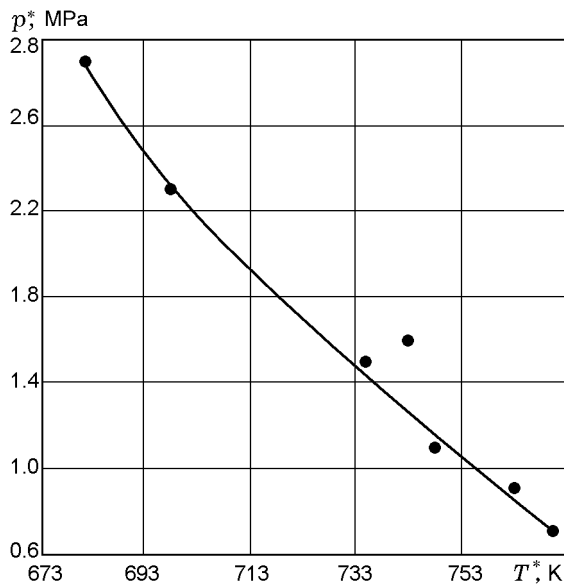**Fig. 1.** Critical ignition pressure of titanium alloys versus the heating temperature of the fractured specimens.

Fig. 2. Critical ignition pressure and maximum temperature of possible heating of juvenile surfaces of VT5-1 alloy specimens in oxygen versus test temperature: points ○ and ● refer to the experimental data of [3] and our calculations, respectively.

For this reason, titanium iodide, characterized by low strength ($\sigma_{\mathrm{ign}} \cong 235$ MPa, $\psi \cong 0.75$, and $S_k \cong 550$ MPa at $T_0 = 293$ K) and, hence, subjected to least heating during fracture compared with other titanium alloys, is extremely resistant to ignition. If titanium is doped with elements that improve its strength properties, juvenile surface fragments are heated to higher temperatures, resulting in a decrease in the oxygen pressure at which these fragments can be ignited.
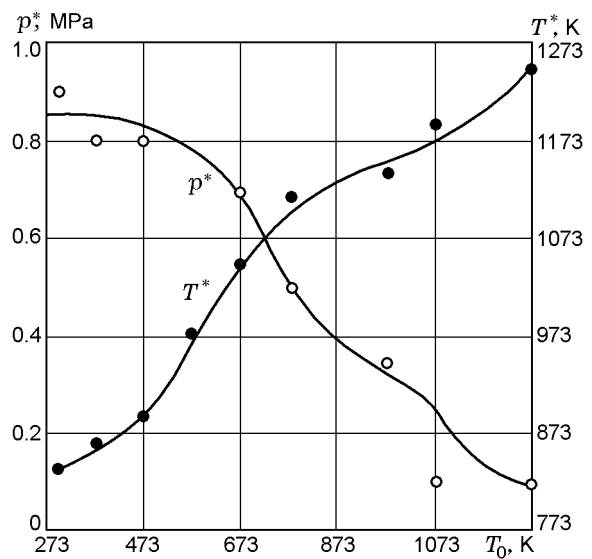
EFFECT OF TEST TEMPERATURE ON CRITICAL PRESSURE

Taking into account the dependence $p^* = f(T^*)$, the effect of the test (ambient) temperature T_0 on the alloy pressure p^* is rather ambiguous because of the dual effect of T_0 on the temperature T^*. An increase in T_0, as a term of the sum $T^* = T_0 + \Delta T$, on the one hand, contributes to an increase in temperature T^*, and, on the other hand, it facilitates a decrease in T^* due to degra-

TABLE 2

Physicomechanical and Thermophysical Properties of VT5-1 Alloy
at Various Temperatures

T_0, K	σ_{ign}, MPa [20]	ψ [19, 20]	S_k, MPa	$\bar{c}_p$, kJ/(kg·K)	ΔT, K
293	863	0.37	1348	0.490	540
373	755	0.41	1242	0.503	488
473	647	0.46	1131	0.545	421
573	608	0.515	1131	0.566	403
673	579	0.550	1119	0.587	376
773	520	0.560	1015	0.628	338
873	392	0.45	754	0.670	232
973	265	0.640	561	0.712	168
1073	147	0.940	402	0.796	114
1173	98	0.950	270	0.800	76

dation of strength properties and fracture work under heating. This results in a decrease in the other term, the heating temperature ΔT. Depending on which of the terms (T_0 or ΔT) changes more significantly, the value of T^* with increase in T_0 can either increase or decrease and the pressure p^* of the materials can decrease or increase, accordingly, as is observed in experiments [9] (see Table 1). For commercial VT1-0 titanium, the pressure p^* increases with increase in temperature T_0 from 293 to 473 K, which is due to a significant decrease in its ultimate strength and, consequently, in ΔT and T^*. At the same time, the strength properties (σ_{ign}) and heating (ΔT) of refractory OT4-1 alloy decrease to a lesser degree with increase in test temperature, which leads to an increase in the total temperature T^* and, hence, a decrease in the alloy pressure p^*.

It is of interest to compare the dependences $T^* = f(T_0)$ and $p^* = f(T_0)$ for alloys at higher test temperatures. Figure 2 and Table 2 illustrate these dependences for VT5-1 alloy, which is one of the few materials for which critical ignition pressures at high temperatures were established experimentally [2, 3]. As can be seen from Fig. 2, there is a clear correlation between the dependences studied: with increase in experimental temperature T_0, the temperature T^* increases and the pressure p^* decreases. In this case, the test temperatures intervals in which T^* and p^* change most rapidly virtually coincide ($T_0 \cong 473$–873 K). Since $T^* = T_0 + \Delta T$ and $\Delta T = f(S_k, \bar{c}_p)$ (2), a comparison of the curves in Fig. 2 and an analysis of the data of Table 2 show that the type of the dependence $p^* = f(T_0)$ for any alloy is determined by the dependence of the strength properties (S_k) and specific heat of this alloy on temperature.

On the other hand, the data of Table 2 show that at nearly room temperatures ($T_0 = 293$ K) at which the strength characteristics are high and the contribution of the heating ΔT to the total temperature T^* is significant ($\Delta T \cong 0.66T^*$), the value of p^* depends largely on ΔT, which, in turn, depends on the strength properties and specific heat of this alloy. Since the latter two characteristics differ greatly, depending on the material, the values of p^* of alloys at relatively low temperatures must also differ greatly, which was confirmed experimentally [2, 9]. At high temperatures ($T_0 = 1173$ K), the value of T^* for alloys is close to the initial test temperature T_0 ($T_0 \cong 0.94T^*$) and depends slightly on the heating ΔT and, hence, on strength characteristics. For this reason, it can be predicted that titanium alloys with similar rates of interaction with oxygen have close values of p^* in high-temperature tests.

As regards the effect of the temperature T_0 on the pressure p^*, we note that in transition to the cryogenic region, the strength characteristics of titanium alloys increase abruptly and their specific heats decrease, which provides for significant potential heating of fractured fragments and, consequently, their ignition. Thus, for example, the ultimate strength of commercial titanium reaches 900 MPa at $T_0 = 90$ K, which leads to an increase in S_k to 2000 MPa and ΔT to 1500 K with preserved satisfactory plasticity ($\psi \cong 0.73$). However, in liquid oxygen, adiabatic heating of elements of a fractured zone is unlikely but, as can be concluded from the fact of titanium self-ignition, the heating under these conditions is sufficient to initiate self-ignition.

EFFECT OF THE GEOMETRICAL DIMENSIONS OF A SPECIMEN AND THE TYPE OF FRACTURE ON CRITICAL IGNITION PRESSURE

The difference in critical ignition pressures between specimens of different shape and cross-sectional areas can be due to the effect of the "scale factor" on the strength and plastic characteristics of the examined specimens, and, hence, on the work of their fracture and heating. Thus, it is known [21] that with decrease in diameter of cylindrical samples from 5 to 1 mm, their strength and plasticity can increase by 10%, which results in a similar increase in the adiabatic heating of juvenile surface fragments and a corresponding decrease in p^*. The work A_{fr} also changes (decreases) during transition from elastic fracture to brittle fracture [21]. For this reason, the temperature T^* is lower for fractured fragments of incised titanium specimens and nitrated or hydrogenated specimens than for flat or initial specimens of the same alloy. This is manifested in an increase in p^*. As was noted in [3], another factor responsible for a rise in pressure p^* during brittle fracturing of titanium alloys is a change in juvenile surface shape during transition from rough fracture to flat fracture. In this process, a decrease in the area subjected to oxidation is accompanied by improvement in heat transfer from fragments of the fracture surface into the depth of the specimen. As a result, these fragments are less heated by the moment of interaction with oxygen than in the case of a rough surface. In the light of the proposed hypothesis, the sites of a juvenile surface that possess an increased ignitability, which are called pyrophoric in the literature [3], can be identified as surface elements heated to high temperature by the work of metal fracture.

EFFECT OF THE TYPE AND RATE OF FRACTURE

It is reasonable to assume that the above consideration of the conversion of plastic strain work to heat during tension of specimens up to rupture is also true for other types of material fracture. Thus, it is known [22] that an impact of a rigid cone on softer metals is accompanied by significant heating of the deformable metal in the indentation zone, whose value is proportional to the hardness of the deformable material. Thus, for soft steel, the heating at room temperature is $\cong 120$ K, and for titanium alloys, which are much harder in comparison with steel, this value is much higher. Therefore, the critical ignition pressure for a titanium alloy frac-

tured by various methods depends on the heat effect accompanying the fracture process and the heat transfer conditions. Since the heat transfer coefficients α differ greatly, the value of p^* is higher, for example, for metal fracturing by an oxygen flow [$\alpha \approx 10^5$ W/(m$^2\cdot$K)] than for the case of tension up to rupture under free convection [$\alpha \approx 10$ W/(m$^2\cdot$K)]: for VT1-0 titanium, $p^* = 6.2$ MPa [8] and 2.3 MPa [9], respectively.

Defining ΔT, we assumed that the heat released due to the work of metal fracture during the formation of a juvenile surface is entirely expended on heating of the elements of this surface with obstructed heat transfer. Naturally, if the rate of formation of the juvenile surface is low, for example, during slow metal fracture, the above conditions are not satisfied and the maximum heating of the elements of this surface will be lower than under adiabatic conditions. This will result in an increase in the pressure p^* of this material. For this reason, slow plastic deformation of specimens of titanium alloys under tensile stresses lower than the ultimate stress does not lead to ignition of the materials even under high pressures (for example, for VT5-1 alloy, $p_{\mathrm{O_2}} \leq 10$ MPa [2]) although a juvenile surface in the form of slip lines is formed under these conditions, too [2]. A low rate of juvenile surface formation is typical of bending tests of titanium plates in high-pressure oxygen, where the metal was ignited only at the moment of fracture [5].

CONCLUSIONS

According to the hypothesis proposed, during titanium fracture in oxygen, a juvenile surface can be ignited only if this surface or its fragments are heated to the ignition temperature T^*; each value of this temperature corresponds to a certain minimum oxygen pressure p^* necessary for the ignition. In this case, the higher T^*, the lower p^*. At low ambient temperatures, the surface can be heated to the required ignition temperature (T^*) by the heat released during metal fracturing. For this, it is necessary that the fracturing process be rapid and the heat transfer from the fractured surface or its fragments be minimal. At room test temperature, the minimal temperature T^* that corresponds to the maximal p^* of alloys (for titanium iodide, $p^* = 7.0$–7.5 MPa) is ≈ 540 K. About half of this temperature is ensured by the heating of juvenile surface fragments due to the work of metal fracture ($\Delta T \approx 245$ K).

We note that although our hypothesis agrees formally with the opinion of Nikolaeva and Zashikhina [10] that a certain temperature corresponds to a certain oxygen pressure at which a free surface is ignited, the term

of "temperature" have different meanings in the formulation of [10] and in the present paper. In[10], this is used to mean test (ambient) temperature T_0). According to our hypothesis, this term means the sum of the temperature T_0 and heating ΔT of juvenile surface fragments due to the work of fracture. Since different alloys are characterized by different works of fracture because of their unequal strength properties, the values of ΔT and $T^* = T_0 + \Delta T$ are also different, which is responsible for the difference in critical ignition pressure between these materials.

To explain the effect of various factors on the ignition ability of alloys, we assume that the chemical interaction of a heated (by the work of fracture) juvenile surface with oxygen proceeds at equal rates and under similar kinetic laws for all the alloys studied. If the interaction patterns are different, as, for example, for α- and β-alloys in oxygen, the values of p^* of the materials differ from one another because of the above factor.

As regards the quantitative relationship between the critical quantities T^* and p^*, it can be established in further investigation of the mechanisms of chemical interaction of heated elements with oxygen. The nature of the effect of oxygen pressure and concentration on the rate of interaction with metal's juvenile surfaces remains to be studied.

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