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# DETERMINING THE THERMOPLASTIC DEFORMATION MECHANISM OF TITANIUM REDUCTION REACTORS AND RECOMMENDATIONS TO INCREASE THE REACTOR SERVICE LIFE

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***The object of research reported in this paper is the stressed-strained state of reactors when producing titanium sponge by the magnesium thermal method, taking into consideration the conditions of their operation and the physical and mechanical properties of the materials.***

***The problem considered is the plastic deformation of the reactor in the process of reducing titanium tetrachloride. To solve this task, an axisymmetric geometric model of the reactor was built using a CAD module of the Comsol Multiphysics software package. For the calculation, the Nonlinear Structural Materials module was used. Owing to the method of finite elements, the critical parameters for the formation of the plastic deformation band of the reactor were determined.***

***Modeling the process of thermoplastic deformation of the reactor under the conditions of obtaining titanium sponge has made it possible to determine the temperature gradient in the upper part of the reactor wall, which leads to local plastic deformation of the wall. The solution to the problem of continuing the reactor service would be to prevent overheating (overcooling) of the reactor wall within the resulting temperature. The physical and mechanical parameters of the material of the reactor wall, necessary to prevent the occurrence of an annular band of plastic deformation, have also been determined. It was shown that at*** $\Delta T>\Delta T_{crit}=60\ ^{\circ}C$***, the walls of a 10-ton reactor during the reduction of titanium tetrachloride with magnesium perceive plastic deformation whose maximum value can reach*** $\varepsilon_{max}^{pl}=5.5\ \%$***.***

***Deformation mechanisms that lead to a change in the shape of the side wall of reactors of magnesium-thermal production of sponge titanium under the action of a heterogeneous temperature field have been determined. The proposed technological solutions are to eliminate local changes in diameter in the upper part of the reactor wall. This will make it possible not only to increase the life of the reactors but will reduce the flow of alloy components into the titanium sponge of nickel, chromium, and iron***

***Keywords: titanium sponge, titanium tetrachloride reduction reactor, reactor thermoplastic deformation process simulation, finite-element method***

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## 1. Introduction

World manufacturers of sponge titanium face a serious problem related to the curvature of reactors in the process of magnesium-thermal production of titanium sponge. This leads to premature damage to reactors and increased production costs [1]. Reactors of the magnesium-thermal process, operating under conditions of sharp heat changes, undergo significant thermal stresses, which prevail over the threshold values of the strength indicators of the material. Solving this problem can significantly increase the efficiency of the metallurgical industry.

Thermal stresses occur at uneven heating or cooling. In technological processes, cooling should be the most uniform. When the reactor is cooled, a temperature gradient occurs in its upper part – the exothermic reaction zone. The stress that occurs in this case depends mainly on the temperature gradient, linear expansion coefficient, and thermal conductivity of the metal. The lower the cooling temperature in the upper part of the retort, as well as the greater the coefficient of linear expansion and the lower the thermal conductivity of the metal, the greater the thermal stress and, correspondingly, the deformation.

Under industrial conditions, the formation of a deformation strip, narrowing of the retort in its upper part are observed, which complicates the unloading of the titanium sponge and shortens the service life of the equipment. In recent years, special attention has been paid to increasing the productivity of the process of magnesium-thermal production of titanium sponge, by increasing the volume of the reactor [2–4]. As a result, the stressed-strained state of the material of the reactor walls is significantly complicated. Increased mechanical stresses and significant temperature gradients are technologically difficult to control. This necessitates a theoretical analysis of the deformation processes in the reactor material. Therefore, building a model of thermoplastic deformation of the material in the reaction zone is extremely relevant and in demand to eliminate the warping of the reactor walls during operation.

## 2. Literature review and problem statement

The main problem in obtaining titanium sponge by the magnesium-thermal method is the warping of the reactor. The reactor is simultaneously affected by a large number of adverse factors. The inner surface is consistently affected by magnesium chloride $MgCl_2$, metallic and liquid magnesium, vapor tetrachloride titanium $TiCl_4$. The outer surface is exposed to high temperature (1000–1200 °C) of the air containing chlorine vapors. The consequence of this effect is the corrosion of the material of the walls of the retort in the oxygen-free oxidizing environment of the furnaces.

Theoretical provisions and causes of deformation of reactors are corrosion in the oxygen-free oxidative medium of reduction and vacuum separation furnaces, which are discussed in [5].

Reactors, as a rule, are made of steel AISI 321 and AISI 304. Steels of this group are analogs of the steel of grade 12X18H10T only when used up to 500–600 °C as they have a different carbon content. In addition, they have an increased content of harmful impurities, such as sulfur and phosphorus, and they also have an increased content of copper. In [5], it is proved that at temperatures above 600 °C, the residual deformation in them increases significantly, which is unacceptable for devices for magnesium-thermal production of sponge titanium operating at 1000 °C. Steels 04X18H10T and AISI 321, despite the low carbon content, on the lower boundary of chromium and nickel, as well as the presence of titanium, are unstable to the interaction of the aggressive environment and are not corrosive in the full sense. Increasing the heterogeneity of the structure of these steels activates corrosion of the inner surface of the reactor, which leads to increased contamination of the titanium sponge with nickel [6]. Attempts to use other grades to increase the service life of the reactors did not lead to a positive result.

The process of shape change, which leads to the failure of reactors, is due to factors related to the consistent effect of the melt of magnesium chloride, metallic and liquid magnesium, vapor-forming titanium tetrachloride loaded into the reactor, on the one hand. And on the other hand, the effect of a temperature of 1000–1200 °C on the walls of the retort with the simultaneous interaction of the hot air of the furnace containing chlorine vapors [3, 4]. As a result of research [7], it was found that the greatest wear of steel 10X18H10T is observed at the boundary of the gas phase with the melt in the zone of the exothermic reaction of titanium tetrachloride with magnesium.

The temperature change in the outer wall of the reactor occurs during the technological process of reducing titanium tetrachloride with magnesium. At the same time, the high temperature of the outer wall of 920–940 °C ensures complete reduction of titanium tetrachloride [8].

The processes of shape change in such an aggressive environment have not been studied enough (as well as the “handling” of the retort material); another issue is the choice of material for the manufacture of retorts [9, 10]. In addition, the optimal dimensions of the retorts and the influence of operating modes are also poorly understood [4].

Modern titanium and magnesium production is characterized by a shortage of mineral raw materials and large volumes of waste; loss of valuable components, which reduces the economic efficiency of production [11].

This suggests that it is expedient to conduct a study aimed at determining the influence of changes in the temperature factor on the warping of the reactor vessel.

The considered problems can be solved by theoretical analysis of deformation processes caused by uneven temperature distribution in the retort reaction zone, and ensuring the minimum temperature of critical overheating of the wall of a 10-ton reactor during the reduction of titanium tetrachloride with magnesium $\Delta T_{crit} = 60$ °C.

## 3. The aim and objectives of the study

The aim of this work is to determine the deformation mechanisms that lead to a change in the shape of the side wall of reactors under the influence of a heterogeneous temperature field. This will make it possible to find technological solutions for the elimination of thermoplastic deformations.

To accomplish the aim, the following tasks have been set:

– to establish the places and magnitude of the greatest development of mechanical stresses in the reactor vessel;

– to determine the minimum overheating temperature of the reactor walls in the reaction zone, which leads to plastic deformation.

## 4. The study materials and methods

The object of study in this work is the stressed-strained state of the reactors, taking into consideration the conditions of their operation and the physical and mechanical properties of the materials. The main hypothesis of the study assumes that the «behavior» of a real reactor can be described on the basis of the «behavior» of its virtual model, built using a CAD module of the Comsol Multiphysics software package.

The retort load diagram and temperature field distribution are shown in Fig. 1.

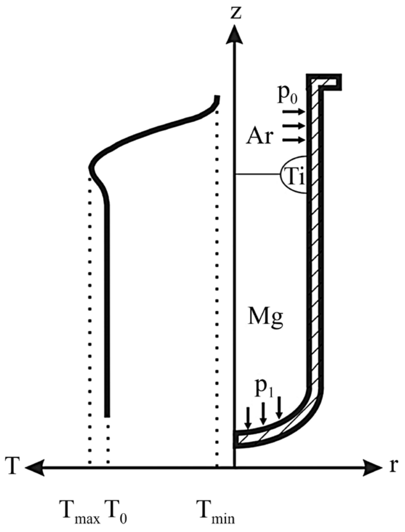
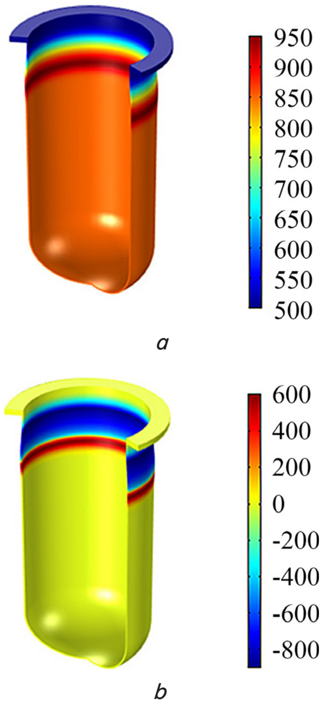


Fig. 1. The scheme of the reactor with the loads acting on it, as well as the distribution of the temperature field on the outer wall. 10-ton reactor with a height of 3.6 m, a diameter of 2.0 m, a wall thickness of 25 mm

A type of reactor model with a temperature field distribution and a temperature gradient are depicted in Fig. 2.



*a*

*b*

Fig. 2. Reactor CAD model: *a* – temperature field distribution $T(z)$, °C; *b* – temperature gradient distribution $\partial T/\partial z$, °C/m

For the calculation, temperature dependences of the reactor material coefficients are used. As a material for the manufacture of retorts, widely used chromium-nickel steel of type AISI 321 was chosen.

The reactor was considered in a cylindrical coordinate system, considering that the displacement vector $u$ does not depend on the angular coordinate $\varphi$ due to the axial symmetry of the reactor:

$$u(r,z)=u_r e_r + u_z e_z. \tag{1}$$

The components of the Cauchy deformation tensor for small movements are determined from the expression:

$$\begin{pmatrix} \varepsilon_{rr} & 0 & \varepsilon_{rz} \\ 0 & \varepsilon_{\varphi\varphi} & 0 \\ \varepsilon_{rz} & 0 & \varepsilon_{zz} \end{pmatrix} = \begin{pmatrix} \partial u_r/\partial r & 0 & (\partial u_r/\partial z+\partial u_z/\partial r)/2 \\ 0 & u_r/r & 0 \\ (\partial u_r/\partial z+\partial u_z/\partial r)/2 & 0 & \partial u_z/\partial z \end{pmatrix}. \tag{2}$$

Complete deformation $\varepsilon$ can be represented as the sum of residual $\varepsilon^0$, elastic $\varepsilon^{el}$, plastic $\varepsilon^{pl}$, and thermal $\varepsilon^{th}$ deformations:

$$\varepsilon=\varepsilon^0+\varepsilon^{\mathrm{el}}+\varepsilon^{\mathrm{pl}}+\varepsilon^{\mathrm{th}}. \tag{3}$$

The tensor of elastic deformations $\varepsilon^{pl}$ is related to the tensor of stresses $\sigma$ via Hooke's law:

$$\sigma_{ij}=C_{ijkl}\cdot\varepsilon_{kl}^{\mathrm{el}}, \tag{4}$$

where $C_{ijkl}$ is an elastic tensor. For an isotropic body, the components of the elasticity tensor can be expressed through the Lamé constants $\lambda$ and $\mu$:

$$C_{ijkl}=\begin{pmatrix} \lambda+2\mu & \lambda & \lambda & 0 & 0 & 0 \\ \lambda & \lambda+2\mu & \lambda & 0 & 0 & 0 \\ \lambda & \lambda & \lambda+2\mu & 0 & 0 & 0 \\ 0 & 0 & 0 & \mu & 0 & 0 \\ 0 & 0 & 0 & 0 & \mu & 0 \\ 0 & 0 & 0 & 0 & 0 & \mu \end{pmatrix}. \tag{5}$$

Lamé constants can be written through the Young modulus $E$ and the Poisson coefficient $v$:

$$\lambda=\frac{\nu E}{(1+\nu)(1-2\nu)},\quad \mu=\frac{E}{2(1+\nu)}. \tag{6}$$

In the zone of plastic deformation, the relationship between stresses $\sigma^{pl}$ and deformations $\varepsilon^{pl}$ is given by the expression:

$$\sigma^{pl}=\sigma_{0.2}+\frac{E_t}{(1-E_t/E)\varepsilon^{pl}}, \tag{7}$$

where $E_t$ is the tangential hardening module; for metals, $E_t$=0.001·$E$.

The condition for the transition from elastic deformation to plastic is the Mises criterion:

$$\sigma_{eff}= =\sqrt{\frac{(\sigma_{rr}-\sigma_{\varphi\varphi})^2+(\sigma_{\varphi\varphi}-\sigma_{zz})^2+(\sigma_{zz}-\sigma_{rr})^2+6\sigma_{rz}^2}{2}}\geq\sigma_{0.2}. \tag{8}$$

Using standard mathematical transformations, we obtain a system of related partial differential equations to determine the components of the displacement vector:

$$\frac{\partial^2 u_r}{\partial r^2}+\frac{\lambda}{\lambda+2\mu}\frac{1}{r}\frac{\partial u_r}{\partial r}-\frac{\lambda}{\lambda+2\mu}\frac{u_r}{r^2}- -\frac{\lambda}{\lambda+2\mu}\frac{\partial^2 u_r}{\partial z^2}=\frac{(3\lambda+2\mu)\alpha}{\lambda+2\mu}\frac{\partial T}{\partial r};$$

$$\frac{\partial^2 u_z}{\partial z^2}+\frac{\lambda}{\lambda+2\mu}\frac{1}{r}\frac{\partial u_r}{\partial z}-\frac{\lambda}{\lambda+2\mu}\frac{\partial^2 u_z}{\partial r^2}=\frac{(3\lambda+2\mu)\alpha}{\lambda+2\mu}\frac{\partial T}{\partial z}. \tag{9}$$

For most materials, the Lamé coefficients $\lambda(T)$ and $\mu(T)$, as well as the temperature coefficient of linear expansion $\alpha(T)$, are nonlinearly dependent on temperature.

To determine the components of the temperature gradient $\partial T/\partial r$ and $\partial T/\partial z$ included in (9), the following analytical relationship was proposed for temperature distribution on the outer wall of the reactor:

$$T(z)=\begin{cases} T_0+(T_{\max}-T_0)\exp\left(-\frac{(z-h)^2}{\delta^2}\right), -H\leq z\leq h; \\ T_{\text{||||}}\quad-(T\quad-T\quad)\times \\ \times\frac{\xi}{\xi+1}\left(\frac{z-h}{H-h-h_0}\right)^n, h\leq z\leq(H-h_0); \\ T_{\min}+\omega(|z-H|)^m,(H-h_0)\leq z\leq H. \end{cases} \tag{10}$$

The exponents of power $n$, $m$ lie in the range of 1.2≤($n$, $m$)≤2.5. $T_0$ is the temperature of the reaction. $T_{\max}$ is the maximum temperature in the exothermic reaction zone. $T_{\min}$ – temperature of the flange cooled by water. $2H$ – reactor height, $H–h$ – distance from the flange to the reaction zone. $h_0$ – distance from the flange to the heaters in the furnace, $\delta$ is the width of the reaction zone. Smoothness of the dependence $T(z)$ is provided by the parameters $\xi$ and $\omega$:

$$\xi = \frac{m}{n}\left(\frac{H-h}{h_0}-1\right), \quad \omega = \frac{1}{h_0^m}\frac{T_{\max}-T_{\min}}{1+\xi}. \qquad (11)$$

We solved (9) by the method of finite elements. For this purpose, on the basis of drawings of real retorts, its axisymmetric geometric model was built using the CAD module of the Comsol Multiphysics software package [12–19]. For the calculation, the Nonlinear Structural Materials module was used. It makes it possible to simulate the behavior of the model during deformation, to determine the zones of elastic and plastic deformation, to predict the destruction of reactors during the process of reduction of spongy titanium.

To simulate the behavior of a 10-ton reactor with a height of 3.6 m, a diameter of 2.0 m, a wall thickness of 25 mm during the recovery process, the following parameters were selected: $T_0$=850 °C, $T_{\min}$=500 °C, $\delta$=0.2 m, $h$=1.0 m, $h_0$=0.3 m. The temperature in the reaction zone $T_{\max}$ ranged from 850 °C to 950 °C. Value $\Delta T=T_{\max}-T_0$ determines the local overheating of the outer wall in the reaction zone of the recovery process, which is automatically cooled from the outside by air from the fans.

## 5. Results of investigating the mechanism of deformation of retorts

### 5. 1. Establishing the place and magnitude of the greatest development of mechanical stresses in the reactor vessel

The temperature distribution over the height of the reactor is shown in Fig. 3.

The dependence of the deformation tensor components is depicted in Fig. 4.

Data analysis in Fig. 3, 4 shows that the distribution of the main (diagonal) components of the deformation tensor is due to the distribution of the temperature field on the side wall of the retort, that is:

$$\varepsilon(z) \approx \varepsilon^{th}(z) = \alpha\left(T(z)-T_{\min}\right). \qquad (12)$$

Distribution profiles of the stress tensor components in the reaction zone for different temperatures are depicted in Fig. 5.

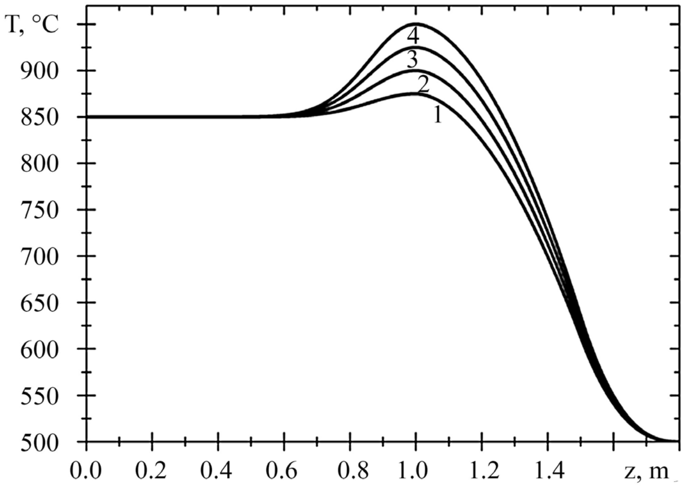


Fig. 3. Temperature distribution in the reaction zone $T(z)$ for different overheating temperatures $\Delta T$: 1 – $\Delta T$=25 °C; 2 – $\Delta T$=50 °C; 3 – $\Delta T$=75 °C; 4 – $\Delta T$=100 °C

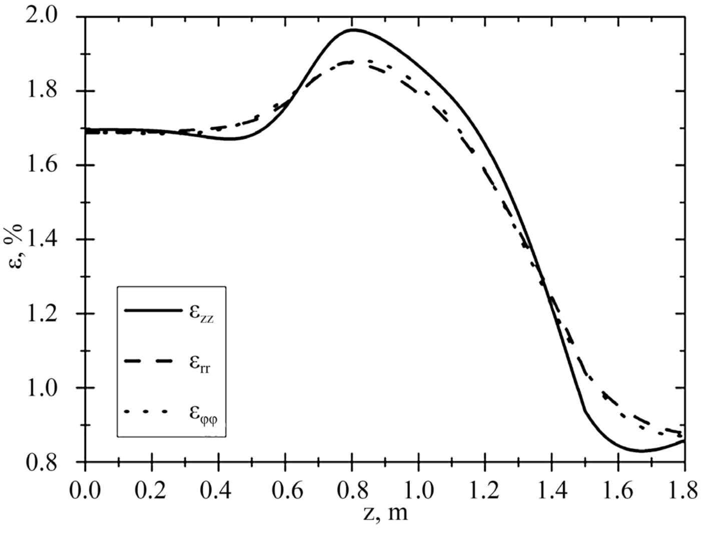
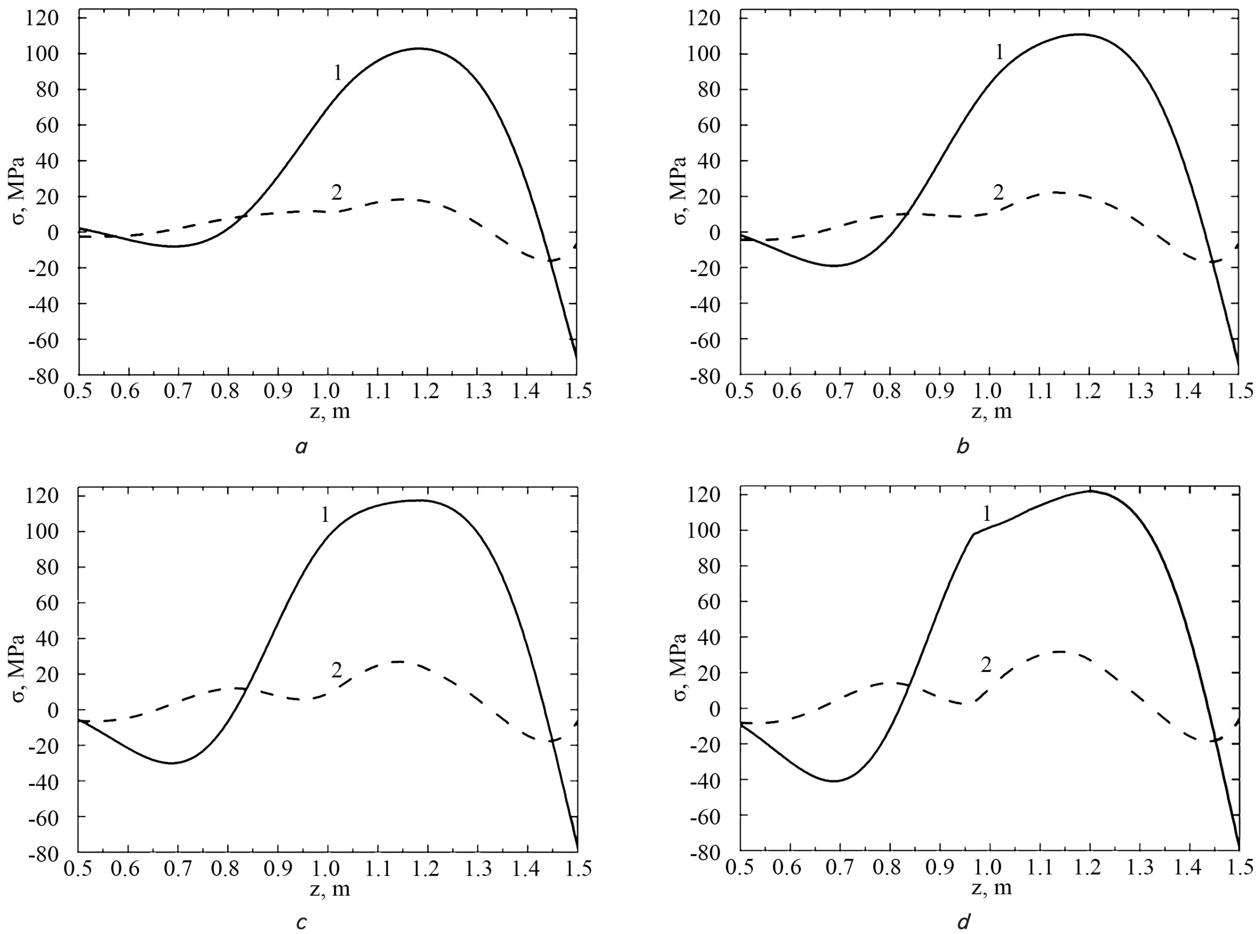


Fig. 4. Distribution profiles of components $\varepsilon_{rr}(z)$, $\varepsilon_{\varphi\varphi}(z)$, $\varepsilon_{zz}(z)$ of a complete deformation tensor $\varepsilon$ calculated at $\Delta T$=100 °C



*a* *b* *c* *d*

Fig. 5. Distribution profiles of the stress tensor component $\sigma_{zz}$ – curve 1 and $\sigma_{\varphi\varphi}$ – curve 2 in the reduction reaction zone for different overheating temperatures $\Delta T$: *a* – $\Delta T$=25 °C; *b* – $\Delta T$=50 °C; *c* – $\Delta T$=75 °C; *d* – $\Delta T$=100 °C

Effective stresses calculated from expression (8) arising in the reduction reaction zone are mainly due to the components $\sigma_{\varphi\varphi}(z)$ and $\sigma_{zz}(z)$ (Fig. 5). The values of the components $\sigma_{rr}$ and $\sigma_{rz}$ are 10–20 times less and do not have a significant impact on the nature of the deformation of the reactors. Local extrema on the dependence $\sigma_{zz}(z)$ correspond to the inflection points $T(z)$. Compressive $\sigma_{ii}<0$ and tensile $\sigma_{ii}>0$ nature of the stresses leads to the curvature of the reactor during the recovery process, that is, to the appearance of a bulge and concaveness of its wall.

### 5. 2. Determining the minimum temperature of plastic overheating of the reactor walls in the reaction zone

Comparing the effective stresses with the conditional yield strength $\sigma_{0.2}$, it is possible to determine the width of the plastic deformation zones $\Delta z^{pl}$ (the area on the $z$ axis where $\sigma_{eff}>\sigma_{0.2}$) and the maximum values of plastic deformation ε↓max*pl*↑ in this area using the expression:

$$\varepsilon_{\max}^{pl} = \max\left(\frac{\sigma_{eff} - \sigma_{0.2}}{E_t}\right). \tag{12}$$

There is a certain temperature range $\Delta T$ for which the condition $\sigma_{eff}<\sigma_{0.2}$ is met (the curve of effective stresses $\sigma_{eff}$ lies below the curve $\sigma_{0.2}$) and there is no plastic deformation (Fig. 6).

With a certain value $\Delta T_{crit}$ in accordance with the criterion of plasticity of Mises $\sigma_{eff}\geq\sigma_{0.2}$, the appearance of plastic deformation is likely. In addition, it follows from Fig. 6 that with an increase in the value of $\Delta T$–$\Delta T_{crit}$, the length of the zones of plastic deformation $\Delta z^{pl}$ and deformation $\varepsilon_{\max}^{pl}$ increase (Fig. 7).

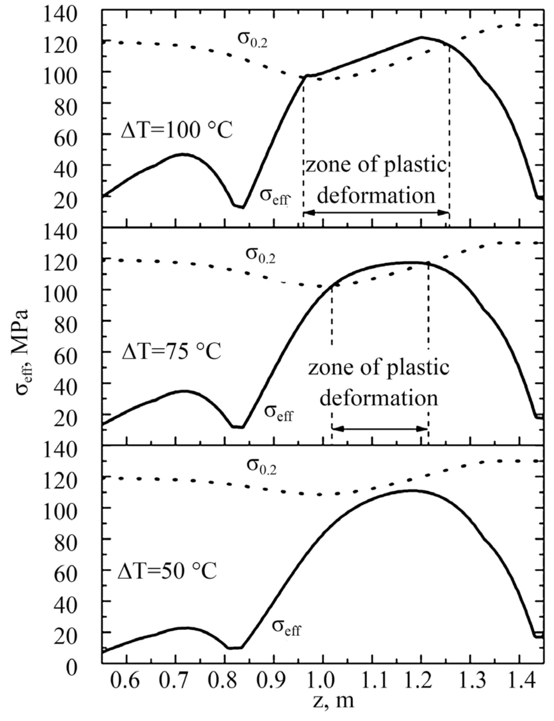


Fig. 6. Distribution of effective stresses according to Mises $\sigma_{eff}$ and conditional yield strength $\sigma_{0.2}$ in the reaction zone at different $\Delta T$

To determine the temperature of critical overheating $\Delta T_{crit}$, $\sigma_{eff}(z)$ were calculated in the temperature range $\Delta T$=50–100 °C in increments of 5 °C and compared with the corresponding dependences $\Delta\sigma_{0.2}(z)$. The resulting dependences $\Delta z^{pl}(\Delta T)$ and $\varepsilon^{pl}_{\max}(\Delta T)$ are shown in Fig. 7.

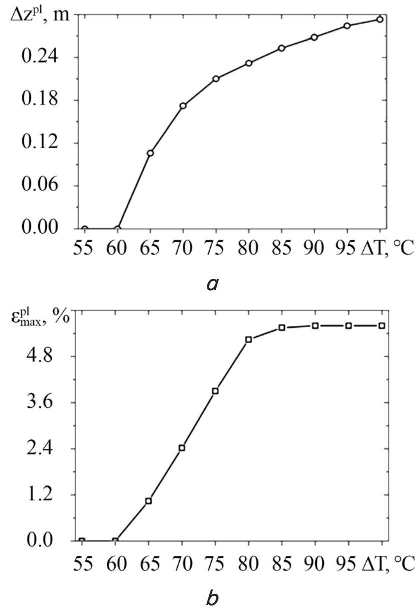


Fig. 7. The dependence of the width of the plastic deformation zone $\Delta z^{pl}$: *a* — on the maximum plastic deformation $\varepsilon^{pl}_{\max}$; *b* — on the overheating temperature $\Delta T$ in the reaction zone

Based on the dependences (Fig. 7), it follows that when $\Delta T > \Delta T_{crit}$=60 °C, the walls of a 10-ton reactor in the process of reducing titanium tetrachloride with magnesium perceive plastic deformation. Its maximum value can reach $\varepsilon^{pl}_{\max} = 5.5\,\%$.

## 6. Discussion of results of investigating the effect of temperature changes in the reaction zone on the process of thermoplastic deformation of the reactor wall

Analytical review showed that the main proposal to solve the problem of thermoplastic deformation of the reactor is to install a refrigerator in the reaction zone, which greatly complicates the structure of the reactor and the technological process of restoring sponge titanium.

Unlike the world's existing reactors, whose performance is 1, 4, and 5 tons per cycle, 10-ton reactors will be subject to more significant temperature drops and corresponding hot deformations. They are just beginning to be introduced into production in the world.

Modeling the process of thermoplastic deformation of the reactor under the conditions of obtaining a titanium sponge made it possible to determine the temperature gradient in the upper part of the reactor wall, which leads to its local plastic deformation. The solution to the problem of increasing the service life of the reactor would be to prevent overheating of the reactor wall within the operating temperature. The physical and mechanical parameters of the reactor wall material, which are necessary to prevent the formation of an annular strip of plastic deformation, have also been determined.

The results of this work allow us to put forward requirements for the use or development of materials and to develop a progressive technology for the reduction of sponge titanium by the magnesium thermal method in high-performance reactors.

The system of equations (9) makes it possible to establish the dependence of the deformation tensor coefficients on the temperature gradient $\partial T/\partial z$. By controlling the temperature gradient, it is possible to influence the plastic deformation of the reactor walls.

The corresponding requirements for the material of reactors have been established, which prevent the appearance of plastic deformation at an operating temperature of 950 °C: the coefficient of linear expansion $\alpha \leq 20 \cdot 10^{-6}$ K$^{-1}$, the conditional yield strength $\sigma_{0.2} \geq 120$ MPa.

Practical use of maintaining temperature restrictions $\Delta T_{crit}$=60 °C makes it possible to increase the life of 10-ton reactors.

The disadvantage of the study is that it does not have a very sensitive temperature control and control system in the reaction zone.

The development of this work is to design a new material that would meet the requirements and create a new temperature control and control system.

## 7. Conclusions

1. The minimum overheating temperature of the reactor walls in the reaction zone, which leads to the appearance of plastic deformation, $\Delta T_{crit}$=60 °C, has been determined.
2. The corresponding requirements for the material of reactors have been established, which prevent the appearance of plastic deformation at an operating temperature of 950 °C: the coefficient of linear expansion $\alpha \leq 20 \cdot 10^{-6}$ K$^{-1}$, the conditional yield strength $\sigma_{0.2} \geq 120$ MPa.

## Conflict of interest

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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