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### Effect of the deformation degree at low temperatures on the phase transformations and properties of metastable austenitic steels

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#### ABSTRACT

**Introduction.** For reliable operation of low-temperature equipment, it is necessary to use materials capable of ensuring operability in a wide temperature range under conditions of alternating loads, exposure to corrosive media, etc. Most often, in such cases, metastable austenitic steels (MAS) of various alloying systems are used. Despite sufficient experience in the use of such materials, not enough information is collected on the behavior of such materials at low temperatures, including phase-structural transformations, the features of such transformations in different temperature zones, including when a load is applied, both static and dynamic. **The subject of the study** in this work is selected MAS 10Cr14NMn20 and 10Cr14Mn14Ni4Ti grades. **The purpose of the study** is to evaluate the performance of industrially used metastable austenitic steels for its possible use instead of steel 12Cr18Ni10Ti. **Research methodology.** The phase composition of the samples was studied on a DRON-3.0 X-ray diffractometer. Mechanical tests were carried out in the temperature range from +20 to –196 °C. Static uniaxial tensile tests were carried out on a R-20 tensile testing machine; cylindrical specimens with threaded heads were prepared according to GOST 11150–75, as well as samples with a circumferential notches. Dynamic bending tests were carried out on a pendulum impact tester, using samples according to GOST 9454–78. **Results and Discussion.** Based on the data obtained, it is found that an increase in the strain rate at low temperatures contributes to a decrease in the number of martensitic phases in the steels under study. It is found that the hardenability during elastic-plastic deformation decreases and completely disappears at the temperature of the material transition to a brittle state. It is shown that an increase in the rate of low-temperature deformation of samples prevents the development of phase martensitic transformations in steels. The results obtained can be recommended for use in the selection of materials for the manufacture of equipment operating at temperatures down to –196 °C. **Conclusions.** It is shown that the obtained values of the characteristics of mechanical properties make it possible to recommend the studied MAS as a substitute for steel 12Cr18Ni10Ti, down to a temperature of –196 °C.

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## Introduction

Low-temperature equipment is widely used in various industries such as metallurgy, chemistry, rocket science, energy and many others. In recent years, such industries as cryobiology, cryomedicine, cryoenergetics have been actively developing, in which it is necessary to use equipment capable of ensuring operability up to temperatures close to absolute zero [1–9].

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It is necessary to use materials that ensure operability at low temperatures for such equipment. These materials should meet such requirements as strength, toughness and plasticity, corrosion resistance, resistance to fracture under abrupt changes in temperature and load application rate [1–4]. In addition, the features of equipment manufacturing should be taken into account: in some cases, it is necessary to manufacture parts by casting, by pressure, besides it is important to take into account the use of welding technologies in the assembly process [4]. Most often, metastable austenitic steels of various alloying systems are used for the manufacture of low-temperature equipment. Despite sufficient experience in the use of such materials, not enough information is collected on the behavior of such materials at low temperatures, including phase-structural transformations, the features of such transformations in different temperature zones, including when a load is applied, both static and dynamic. There is a lack of information about the effect of stress concentrators, which always appear in equipment parts at different stages of its manufacture, on changes in the physical and mechanical properties of metastable austenitic steels [1–3, 5–20].

In order to make a conclusion about the possibility of using materials at low temperatures, including cryogenic one, it is necessary to conduct studies to assess changes in the structure and complex of properties of steels during cooling, including at abrupt temperature changes, for example, in the process of filling containers with a liquid cryoproduct. An analysis of the structure and properties of materials after long-term operation of low-temperature equipment made it possible to conclude that the traditionally used set of studies for choosing a material is, as a rule, insufficient and cannot guarantee reliable operation of equipment. This is due to the fact that numerous technological heatings during long-term operation can lead to changes in the phase-structural composition. This, in turn, can be the cause of emergency situations, leading to premature destruction of low-temperature equipment, for example, tanks and pipelines [5, 21–24].

In this regard, it is important to obtain information about the change in the phase-structural state and mechanical characteristics of metastable austenitic steels traditionally used in low-temperature equipment. The collected information on the behavior of materials will make it possible to clarify the recommendations for choosing a material for low-temperature equipment, including cryogenic one, as well as its reliability during long-term operation.

**The purpose of the study** is to evaluate the performance of industrially used metastable austenitic steels (*MAS*) for its possible use instead of steel *12Cr18Ni10Ti*.

**Study objectives:**

- to evaluate the joint effect of low temperatures and deformations on the processes of phase-structural transformations in metastable austenitic steels of various alloying systems;
- to investigate the influence of the manufacturing method (cast or deformed state), the presence of stress concentrators, the rate of load application and temperature changes on the properties of austenitic steels;
- to give an opinion on the possibility of replacing the traditionally used steel *12Cr18Ni10Ti* for the manufacture of equipment for low-temperature equipment, including cryogenic one.

### Research technique

Traditionally used metastable austenitic steels of *Cr-Ni-Mn* and *Cr-N-Mn* alloying systems were chosen as objects of study. The chemical composition of the industrial casts of the studied steels is given in Table. The composition was determined by the X-ray spectral method.

**Chemical composition of steels *10Cr14NMn20*, *10Cr14Mn14Ni4Ti***

Steel grade	Chemical element, wt. %									
	<i>C</i>	<i>Si</i>	<i>Mn</i>	<i>Ni</i>	<i>S</i>	<i>P</i>	<i>Cr</i>	<i>N</i>	<i>Ti</i>	<i>Cu</i>
<i>10Cr14NMn20</i>	0.10	0.5	20.3	–	0.011	0.012	14.8	0.3	0.45	–
<i>10Cr14Mn14Ni4Ti</i>	0.10	0.7	14.9	–	0.019	0.020	14.6	–	0.23	–

The steels were smelted in an induction steel furnace. The resulting ingots were forged into blanks with a cross section of  $30 \times 40$  mm and a diameter of 20 mm. The forging temperature was chosen to be 1,000–1,250 °C. The resulting workpieces were subjected to heat treatment, consisting of austenitization for steel *10Cr14NMn20* at a temperature of 900–950°C and 1,000–1,050°C for steel *10Cr14Mn14Ni4Ti* with cooling in water.

The phase composition of the samples was studied on a *DRON-3.0* X-ray diffractometer. Mechanical tests were carried out in the temperature range from +20 to –196 °C. Static uniaxial tensile tests were carried out on a *R-20* tensile testing machine; cylindrical specimens with threaded heads were prepared according to GOST 11150–84 “Metals. Methods of tension tests at low temperatures”, as well as samples with a circumferential notches. Dynamic bending tests were carried out on a pendulum impact tester, using samples according to GOST 9454–78 “Metals. Method for testing the impact strength at low, room and high temperature”.

## Results and discussion

It is known that in metastable austenitic steels, phase transformations can occur during the manufacture of products using forging, stamping, and other types of impact, as well as during operation under dynamic loading conditions at low temperatures. Taking into account the degree of responsibility of low-temperature equipment, a set of studies was carried out to determine the dependence of martensitic transformations in industrially used metastable austenitic steels *10Cr14NMn20* and *10Cr14Mn14Ni4Ti* on the strain rate and test temperature.

In the process of analyzing phase transformations occurring under deformations and low temperatures in steel *10Cr14NMn20* at different strain rates, the following was revealed. When steel *10Cr14NMn20* is deformed at 20 °C at a rate of  $\dot{\epsilon} = 0.34 \times 10^{-4} \text{ s}^{-1}$ ,  $\epsilon$ -martensite is formed immediately. At the same time, an increase in the rate to  $\dot{\epsilon} = 0.34 \times 10^{-1} \text{ s}^{-1}$  causes the formation of  $\epsilon$ -martensite only after deformation by 25 %, and at a rate equal to  $\dot{\epsilon} = 0.34 \times 10^2 \text{ s}^{-1}$ , the solid solution remains stable up to the destruction of the samples (Figure 1).

Lowering the test temperature to –100 °C and further to –196 °C is accompanied by the appearance of  $\alpha$ -martensite. Characteristically, at –100 °C,  $\alpha$ -martensite appears after 10–15 % strain, and its amount increases with further deformation. The amount of  $\epsilon$ -martensite under these conditions first increases and

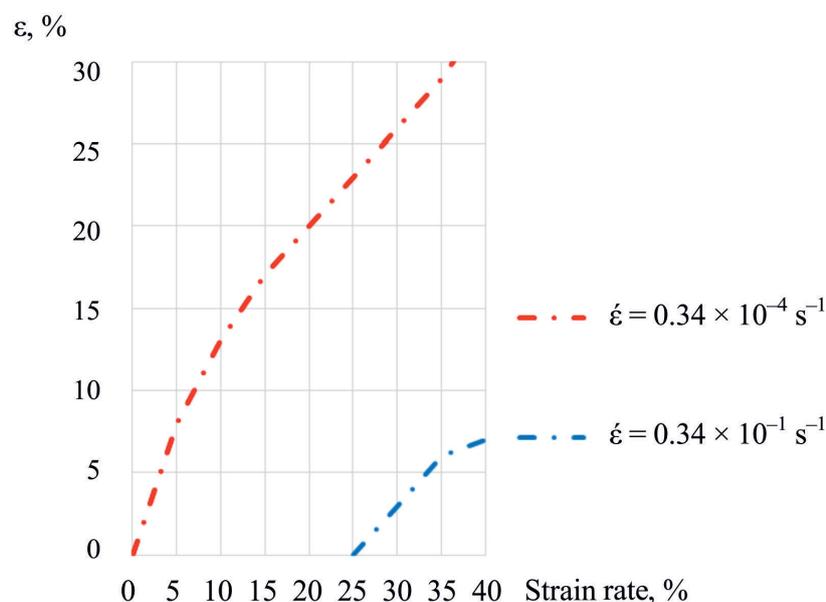


Fig. 1. Dependence of martensitic transformations in steel *10Cr14NMn20* on the strain rate at a test temperature of 20 °C

then decreases (Figure 2). This may indicate that phase transformations proceed in the sequence  $\gamma \rightarrow \varepsilon \rightarrow \alpha$ . It is also characteristic that an increase in the rate by a factor of 103 reduces the degree of decomposition of the  $\gamma$ -solid solution.

Martensitic transformations of the  $\gamma$ -solid solution at  $-196\text{ }^\circ\text{C}$  and the same rates occur similarly as at  $-100\text{ }^\circ\text{C}$ , except that the joint appearance of  $\varepsilon$ - and  $\alpha$ -martensite is observed (Figure 3).

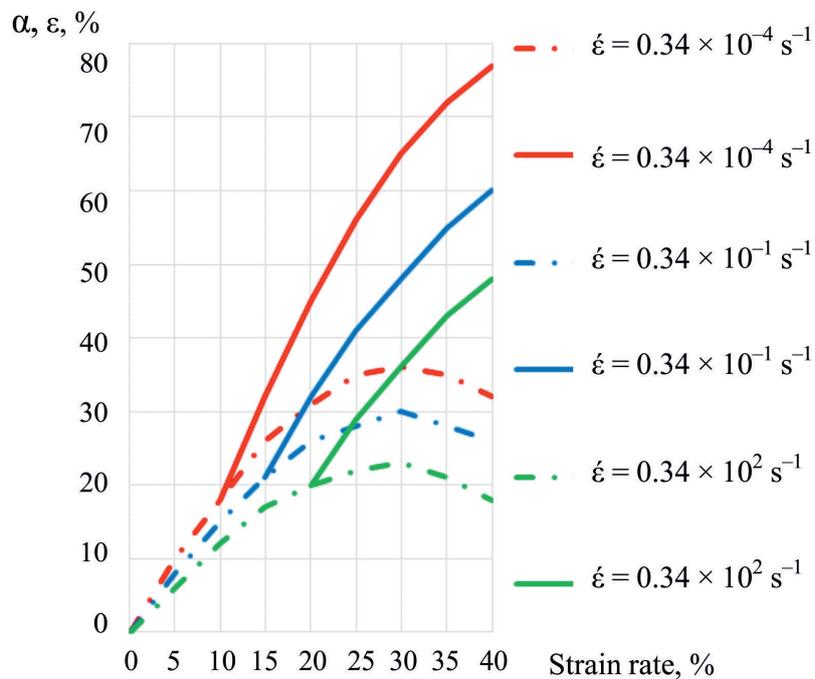


Fig. 2. Dependence of martensitic transformations in steel *10Cr14NMn20* on the strain rate at a test temperature of  $-100\text{ }^\circ\text{C}$

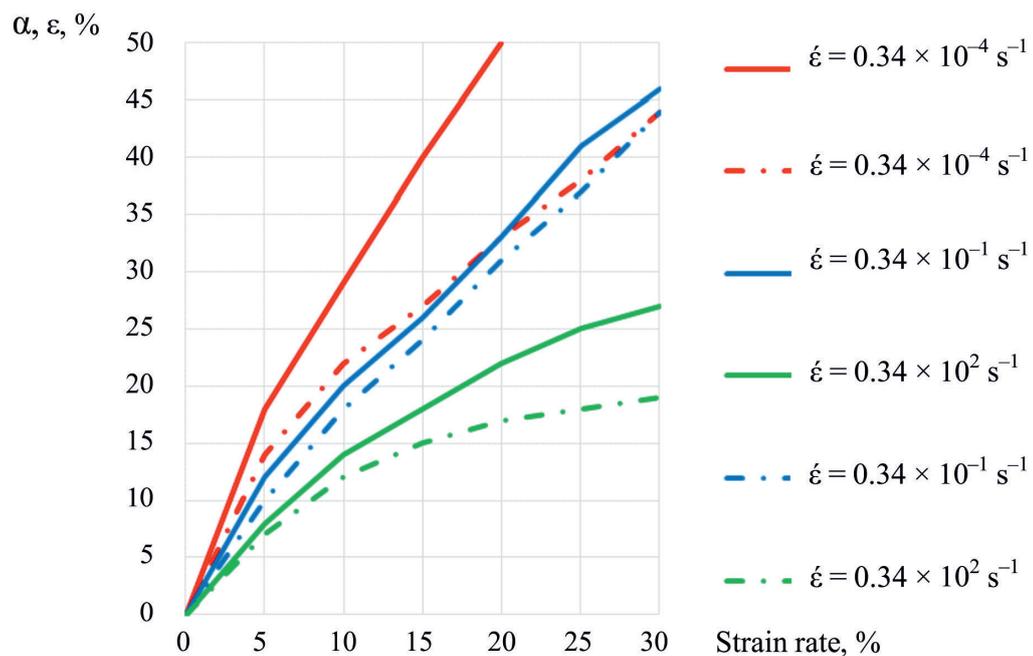


Fig. 3. Dependence of martensitic transformations in steel *10Cr14NMn20* on the strain rate at a test temperature of  $-196\text{ }^\circ\text{C}$

Thus, it has been established that an increase in the strain rate reduces the degree of transformation of austenite into martensite and does not affect its kinetics.

When analyzing phase transformations occurring under the influence of deformations and low temperatures in steel *10Cr14Mn14Ni4Ti* at different strain rates, the following was revealed.

As a result of cooling below 20 °C, two martensitic phases  $\alpha$ - and  $\varepsilon$ -martensite appear. As the temperature decreases, the amount of these phases increases, but does not exceed 12 % for  $\varepsilon$ -martensite and 8 % for  $\alpha$ -martensite (Figure 4).

During low-temperature deformation of steel *10Cr14Mn14Ti*, as the temperature decreases, the amount of austenite and  $\varepsilon$ -martensite decreases, and  $\alpha$ -martensite increases. It should be noted that in the temperature range of deformation from 20 °C to -100 °C, the intensity of formation of  $\alpha$ -martensite is low and, apparently, in this temperature range, the transformation occurs according to the scheme  $\gamma \rightarrow \varepsilon \rightarrow \alpha$ , and with a further decrease in temperature, the amount of  $\alpha$ -phase increases sharply. With an increase in the strain rate, the transformation of austenite into martensite decreases (Figure 5).

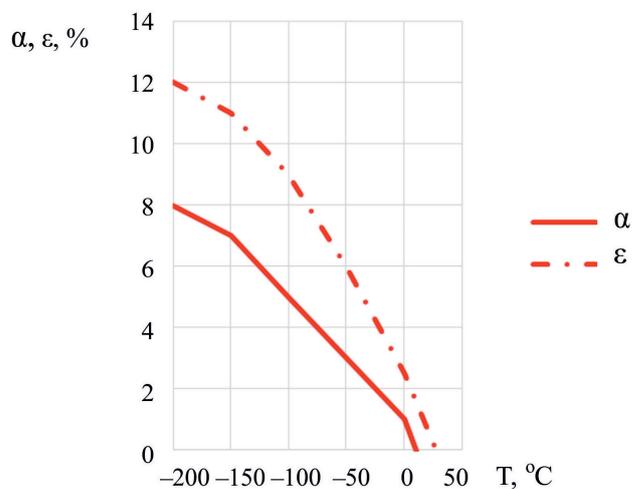


Fig. 4. Dependence of martensitic transformations in steel *10Cr14Mn14Ti* on test temperature

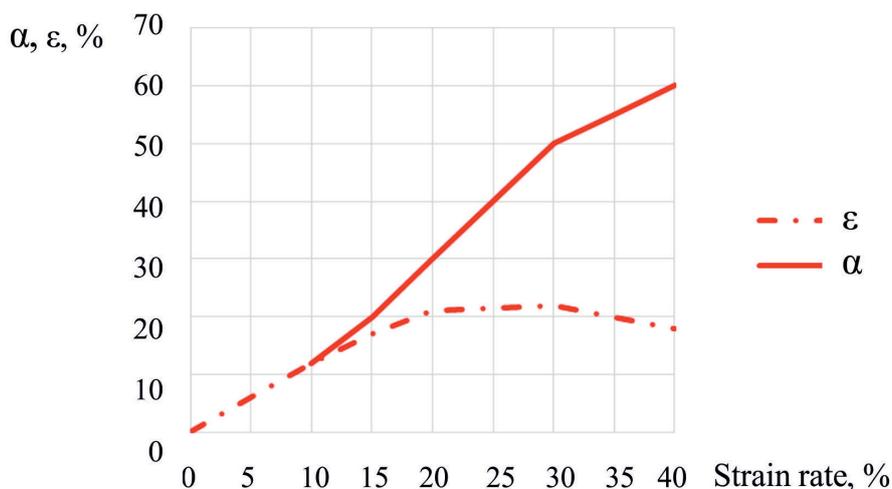


Fig. 5. Dependence of martensitic transformations in steel *10Cr14Mn14Ni4Ti* on the strain rate at a test temperature of -196 °C

It is shown that a decrease in the amount of secondary structural phases  $\alpha$ - and  $\varepsilon$ -with an increase in the strain rate of metastable austenitic steels can be associated with sample heating.

The authors of [4, 13–17] showed that high-speed deformation leads to a sharp increase in temperature on the shear planes. Areas of high-temperature heating, as a rule, are grouped in thin shear layers, as a result neighboring zones are heated slowly. It follows that the decrease in the amount of martensitic phases associated with an increase in the strain rate can be explained by an increase in the temperature of the samples due to the heat released during deformation. At the same time, an increase in the proportion of the  $\alpha$ -phase with an increase in the strain rate may be due to the fact that the samples were stretched under isothermal conditions. Thus, during isothermal tension, an increase in the strain rate leads to an increase in the proportion of  $\alpha$ -martensite formed, and when the sample is heated, it prevents the formation of secondary structural phases.

The paper analyzes the influence of low temperatures and stress concentrators on the value of the ultimate strength of the studied steels in the deformed and cast state. It has been established that with decreasing temperature, the value of ultimate strength increases. The stress concentration caused by the circumferential notch led to a more significant increase in the ultimate strength characteristics, especially with decreasing temperature. It should be noted that the studied steels favorably differ from the traditionally used  $12Cr18Ni10Ti$  [4] by a higher level of ultimate strength over the entire temperature range. In addition, a comparison of the properties of the cast and deformed state showed that in the deformed state both steels have higher values of ultimate strength (Figure 6).

The paper analyzes the effect of low temperatures and stress concentrators on the value of the yield strength of the studied steels in the deformed and cast state. From the studies carried out, it can be seen that with a decrease in temperature, the value of the ultimate strength increases. The stress concentration caused by the circumferential notch led to a more significant increase in the yield strength, especially as the temperature was lowered. It should be noted that the studied steels favorably differ from  $12Cr18Ni10Ti$

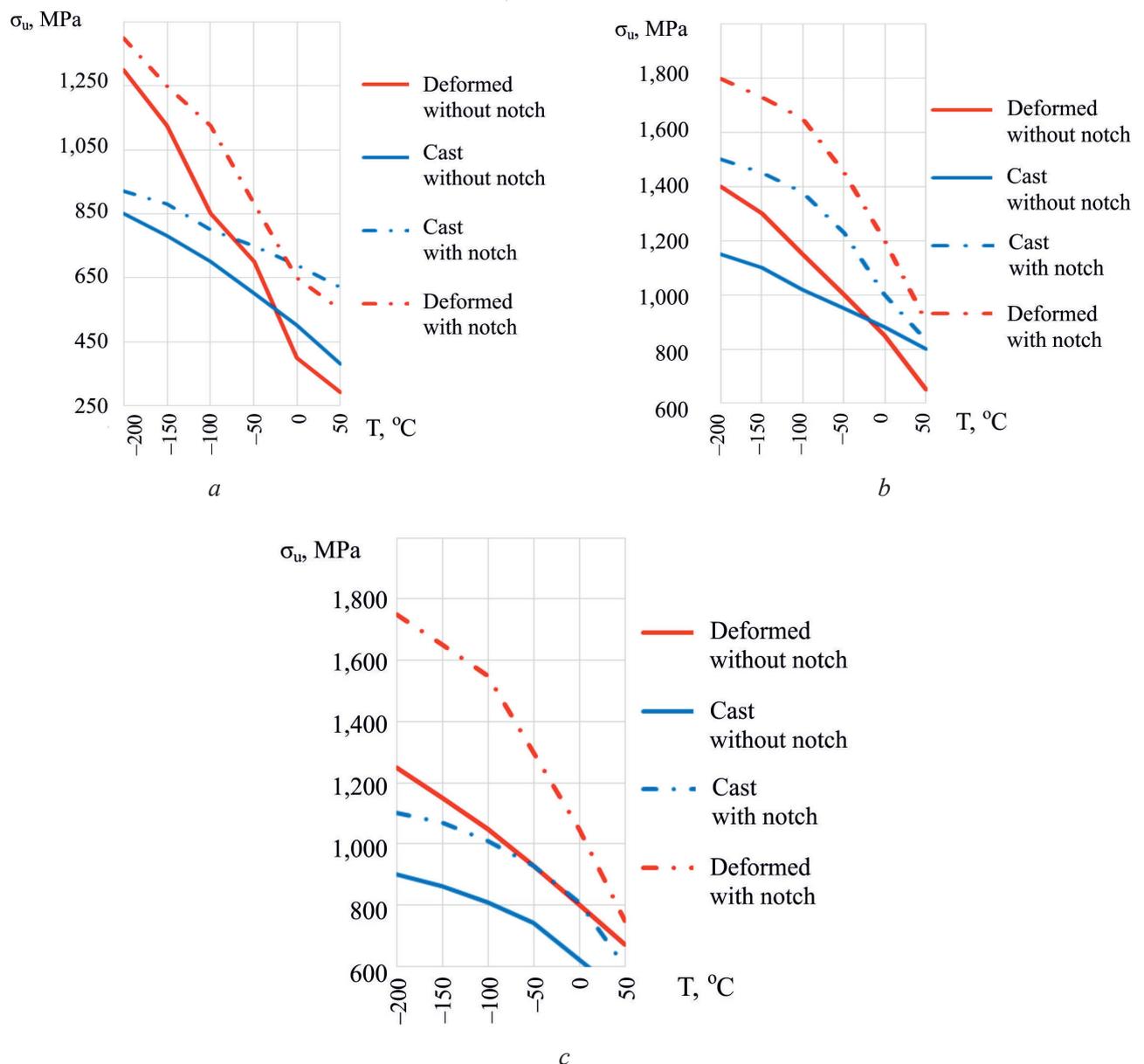


Fig. 6. The influence of low temperatures and the presence of stress concentrators on the value of the ultimate strength of steels:

*a* –  $12Cr18Ni10Ti$  [4]; *b* –  $10Cr14Mn20$ ; *c* –  $10Cr14Mn14Ni4Ti$

by a higher level of yield strength, and also that steels in the deformed state have higher values of the yield strength (Figure 7).

The paper analyzes the influence of low temperatures and stress concentrators on the value of the percentage reduction of area of the studied steels in the deformed and cast state. The value of the percentage reduction of area on samples with a notch is significantly lower than without it. It is known [4] that a notch hinders the development of plastic deformation, which begins at its tip, since the proportion of tangential stresses sharply decreases from the notch to the center of the sample. The influence of a sharp deep notch is manifested for all steels, regardless of its strength level, type of crystal lattice, toughness and plasticity.

It is established that according to the characteristics of the percentage reduction of area, steel  $10Cr14Mn14Ni4Ti$  is not inferior to steel  $12Cr18Ni10Ti$  (Figure 8). The data for steel  $10Cr14NMn20$  are at an acceptable level, although for the cast condition values of the percentage reduction of area is somewhat lower than those for steel  $12Cr18Ni10Ti$ .

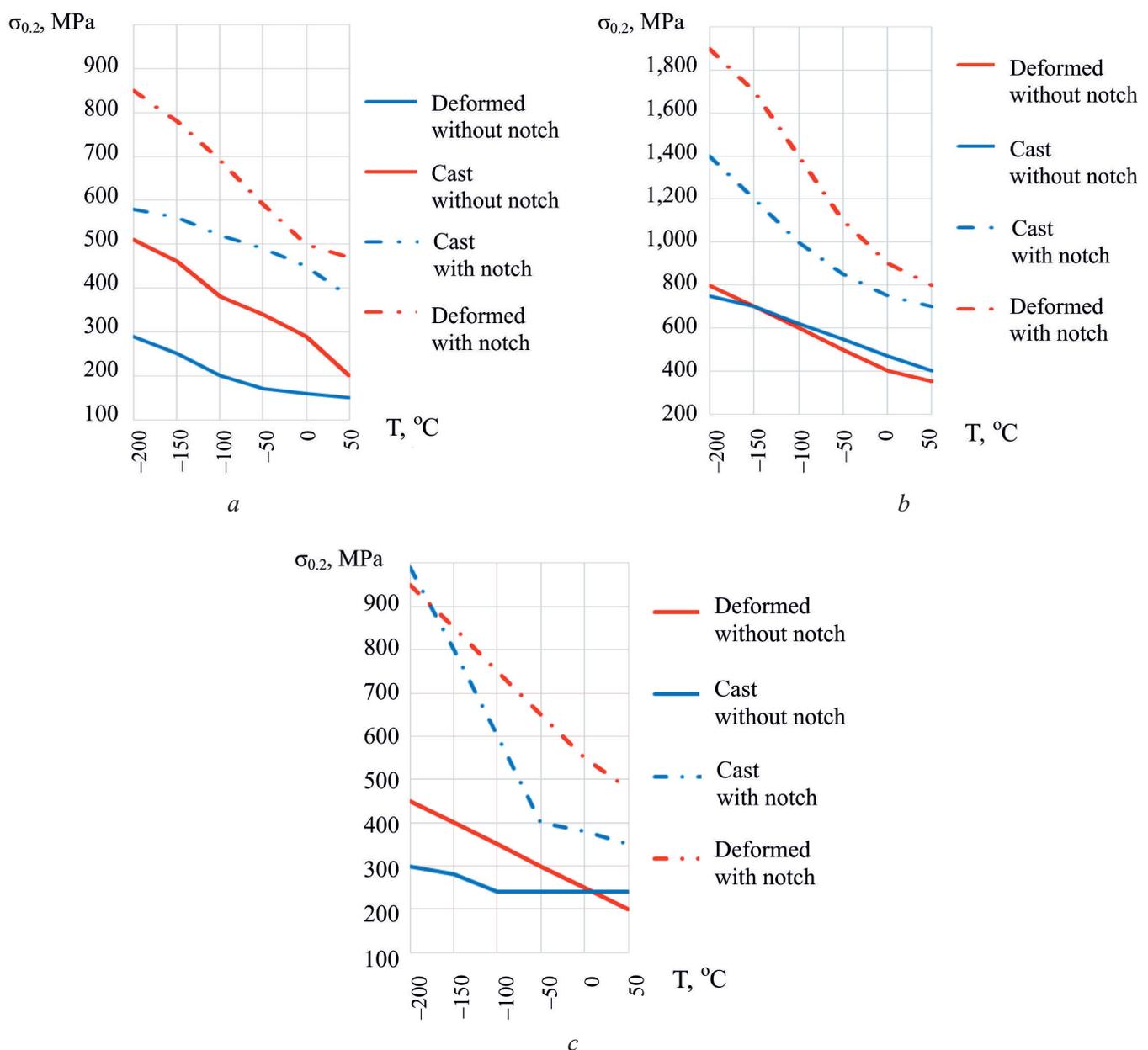


Fig. 7. The influence of low temperatures and the presence of stress concentrators on the value of the ultimate strength of steels:

a –  $12Cr18Ni10Ti$  [4]; b –  $10Cr14NMn20$ ; c)  $10Cr14Mn14Ni4Ti$

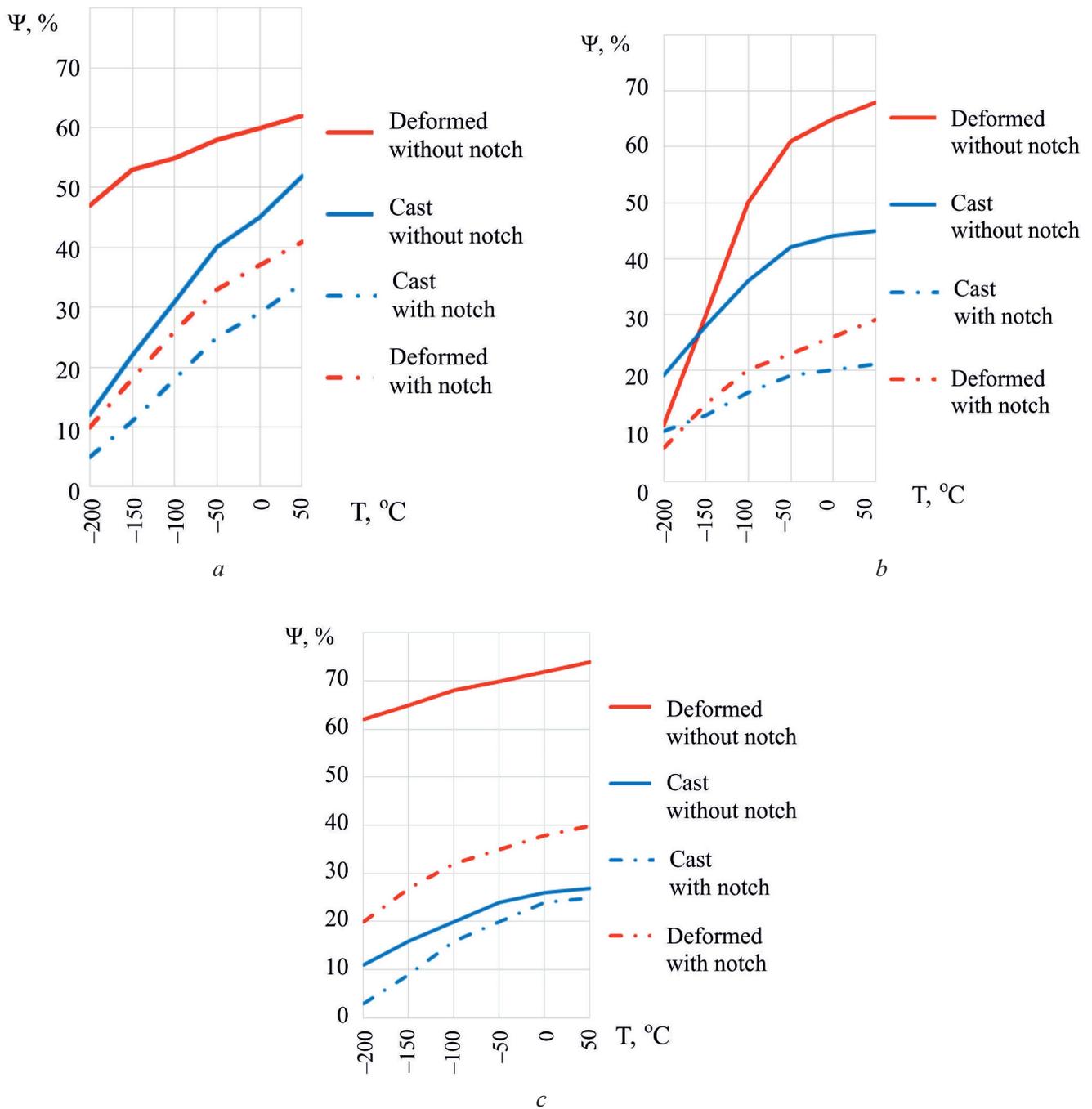


Fig. 8. Effect of low temperatures and the presence of stress concentrators on the percentage reduction of area of steel:

*a* – 12Cr18Ni10Ti [4]; *b* – 10Cr14Mn20; *c* – 10Cr14Mn14Ni4Ti

Taking into account the difficult operating conditions of low-temperature equipment materials, the work analyzes the effect of low temperatures and stress concentrators on the values of impact strength of the studied steels in the deformed state (Figure 9).

A comprehensive analysis has shown that 10Cr14Mn20 steel and 10Cr14Mn14Ni4Ti steel in a deformed state are characterized by a sharp reduction of the values of impact strength in a small temperature range. At the same time, it is important to maintain sufficiently high values of impact strength at a test temperature of  $-196$  °C.

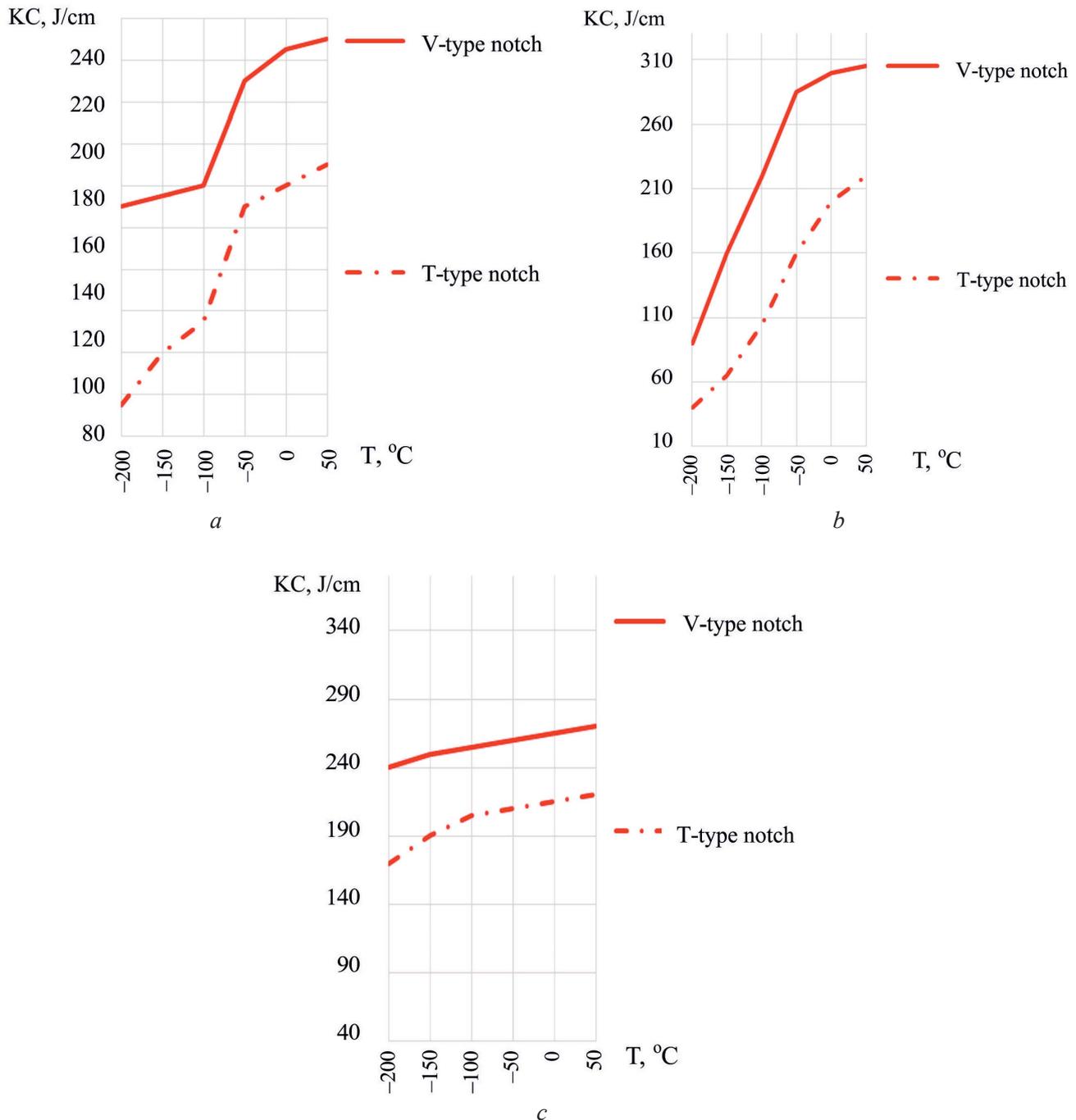


Fig. 9. Effect of low temperatures and the type of stress concentrator on the values of impact strength of steels in the deformed state:

*a* – 12Cr18Ni10Ti [4]; *b* – 10Cr14NMn20; *c* – 10Cr14Mn14Ni4Ti

## Conclusion

It was found that an increase in the strain rate from  $0.34 \times 10^{-4} \text{ s}^{-1}$  to  $0.34 \times 10^{-1} \text{ s}^{-1}$  and further to  $0.34 \times 10^2 \text{ s}^{-1}$  at temperatures below  $0 \text{ }^{\circ}\text{C}$  contributes to a decrease in the number of martensitic phases in the studied steels.

It was confirmed that the presence of a stress concentrator on cylindrical samples in the deformed and cast state during static tensile testing provided an increase in strength values, with a decrease in the values of toughness and plasticity.

It was found that the hardenability during elastic-plastic deformation decreases and completely disappears at the temperature of the material transition to a brittle state;

It was experimentally determined that for metastable austenitic steels, the fracture energy of samples under static bending turned out to be less than under dynamic bending. An increase in the rate of low-temperature deformation of samples prevents the development of phase martensitic transformations in steels.

It was established that the obtained values of mechanical properties characteristics make it possible to recommend the studied metastable austenitic steels as a substitute for the widely used austenitic steel *12Cr18Ni10Ti* up to a temperature of  $-196\text{ }^{\circ}\text{C}$ , both for the deformed and for the cast state.

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## Conflicts of Interest

The authors declare no conflict of interest.

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