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ASSESSMENT OF HYDROGEN EMBRITTLEMENT IN HIGH-ALLOY CHROMIUM-NICKEL STEELS AND ALLOYS IN HYDROGEN AT HIGH PRESSURES AND TEMPERATURES

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The existence of two (low-and high-temperature) extremes of hydrogen embrittlement in heat-resistant austenitic steels and alloys with intermetallic hardening in the range of 293–1073 K was revealed. The low-temperature minimum of their properties in hydrogen is 250–300 degrees higher than that of martensitic and homogeneous austenitic steels. The high-temperature maximum of hydrogen embrittlement manifests itself at 1073 K in steels and alloys with intermetallic hardening and a small percentage of refractory elements (Mo, Nb, W), which retard phase transformations during tests. At 293 K, the action of the external hydrogen atmosphere and absorbed internal hydrogen is determined by the structural class and the nickel content of the material. The degree of brittleness of nickel-base alloys (56 and more wt.% Ni) and heat-resistant martensitic steels is determined by the gaseous hydrogen pressure, and the additional action of internal hydrogen is perceptible only at low pressures. The ductility and low-cycle life characteristics of austenitic steels (23–28 wt.% Ni) deteriorate only after hydrogen presaturation and change only slightly with increasing hydrogen atmosphere pressure, and iron-nickel alloy (43 wt.% Ni) is sensitive to the action of external and internal hydrogen. The existence of a hydrogen degradation limit, the limiting minimum values of the performance characteristics of steels and alloys (specific elongation and lateral contraction ratio, number of cycles to fracture), which do not decrease with increasing adsorbed hydrogen pressure and absorbed hydrogen content and with decreasing loading rate and frequency, has been established. Such values of the mechanical characteristics of martensitic steels and nickel-base alloys are achieved at hydrogen pressures of over 10 and 30 MPa and of dispersion-hardening austenitic steels and alloys at a hydrogen content of 15 and 30 ppm, respectively.

Keywords: short-term strength and ductility, low-cycle life, heat-resistant chromium-nickel steels and alloys, hydrogen embrittlement.

Introduction. The hydrogen embrittlement of structural materials is one of the important problems of the physicochemical mechanics of materials. Its essence is that in a hydrogen environment, metals lose their ductility, and a brittle fracture occurs at much lower loads than in an inert environment [1–6]. Depending on the structural state of the material, the method and type of loading (short-term or long-term, static or cyclic loading), hydrogen environment characteristics and temperature, hydrogen can differently affect the structural strength of metallic products.

The degree of the hydrogen embrittlement of metals depends on the following parameters: the shape and size of the specimen, type and rate of loading, hydrogen pressure, temperature and purity of the hydrogen atmosphere. In the case of non-identity of one or several of these factors, different, sometimes contradictory, results can be obtained

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[4–8]. In spite of many years of research, the uniform methodological requirements for experiments in a hydrogen environment, e.g., on short-time tension and low-cycle fatigue, have not been formulated up to now [7–12].

The operating conditions of hydrogen power equipment envisage static and cyclic loads on components over a wide temperature range [1–4, 7–12], and the data, known from publications, on the temperature dependences of mechanical characteristics in hydrogen are limited and ambiguous. According to [1, 2, 4, 5], the upper embrittlement temperature of steels in a hydrogen environment under short-time static tension and low-cycle fatigue is 573 K. At the same time, our studies and some studies by other authors revealed a significant decrease in the ductility characteristics of heat-resistant nickel steels by the action of hydrogen at 1073 K and a pressure of 35 MPa [1, 2, 10–14]. Besides, the influence of hydrogen, absorbed by the material during high-temperature operation, at room temperature is scantily studied.

This paper presents results of studies of the effect of temperature, pressure and hydrogen content on the short-term strength, ductility and low-cycle life of steels and nickel-base alloys in hydrogen-containing gaseous environments in a wide pressure and temperature range.

Materials and Research Procedure. We investigated materials from which components are made which are used in power engineering, petrochemical engineering and aerospace products [3–5, 14, 15] for operation in a hydrogen environment. The chemical composition, heat treatment conditions and mechanical properties at 293 K in helium (hydrogen-unsaturated specimens) and hydrogen at a pressure of 35 MPa (specimens with a hydrogen content of up to 5 ppm, 15Kh12N2MVFAB steel; 15 ppm, 10Kh11N23T3MR and 10Kh15N27T3MR steels; 19 ppm, KhN43BMTYu alloy; 24 ppm, KhN55MBYu alloy) are listed in Table 1. Structurally, they are 15Kh12N2MVFAB martensitic steel with a retained austenite content after optimal heat treatment of about 10%; dispersion-hardening austenitic steels and alloys: 10Kh11N23T3MR, 10Kh15N27T3MR, KhN43BMTYu, and KhN55MBYu with a different ratio of iron content to nickel content, which are hardened with carbides and mainly with an intermetallic γ'-phase of the $(Ni, Fe, Cr)_3$ (Al, Ti, Nb) type in a total amount of up to 6% (10Kh11N23T3MR steel), 10% (10Kh15N27T3MR steel and KhN43BMTYu alloy) and 8% (KhN55MBYu alloy).

Standard 25 mm-long smooth cylindrical specimens with a test portion diameter of 5 mm were subjected to a static tension test with a velocity of travel of the active grip of 10^{-2}–10^{2} mm/min. The low-cycle life (number of cycles to fracture, N) under stiff pure pulsating bending was determined over a pressure range of 0–35 MPa at an amplitude of 1.6% with a frequency of 0.5 Hz on ground flat specimens with a $3 \times 6 \times 20$ mm test portion. The tests were conducted according to GOST 9651-84 (ISO 783-89) and GOST 25.502-79 [16, 17].

Some specimens were held for 10 h in a hydrogen atmosphere at 623 K and 35 MPa; hydrogen-saturated and hydrogen-unsaturated specimens were tested in helium and hydrogen at a pressure of 35 MPa. The volumetric hydrogen content of the metal was determined by the extraction method and with the aid of a Leko TCH 600 device. The sensitivity to hydrogen degradation was assessed from the coefficient β, which was determined as the ratio of the values of corresponding characteristics in hydrogen and helium, e.g., $\beta_\psi = \psi_H / \psi_{He}$ is the coefficient of influence of hydrogen on lateral contraction ratio, which is most sensitive to the action of hydrogen under short-time static tension [1, 2, 12].

Assessment of the Effect of Loading Conditions on the Hydrogen Embrittlement of Materials at Room Temperature. As the static extension rate decreases, the action of the hydrogen atmosphere or preabsorbed internal hydrogen is first enhanced, and after reaching the critical rate, the properties are stabilized (Fig. 1). Under the same conditions of hydrogen action, the rate range of the maximum embrittlement of martensitic steel is two orders of magnitude wider than that of austenitic steel, which correlates with the ratio of the diffusion coefficients in materials with body- and face-centered lattices [2, 6, 8] (Fig. 1).

All further static tension tests were carried out at a loading rate of 0.1 mm/min.

The effect of hydrogen pressure and content on mechanical properties was studied to determine the conditions of experiments, which will enable comparative evaluation of the performance of materials of different structural classes and methods of hardening in hydrogen. It is known [1, 2, 8, 10–12] that the effect of hydrogen on mechanical characteristics increases in proportion to the square root of pressure almost for all materials, the range of this dependence being determined by the chemical composition and structure of the material. Under short-time static

TABLE 1. Chemical Composition, Heat Treatment Conditions and Mechanical Properties of Alloys at Room Temperature in Air (above the line) and in Hydrogen under a Pressure of 35 MPa after Hydrogen Presaturation (below the line)

Material chemical composition (wt.%)	Heat treatment		σ_u, MPa	$\sigma_{0.2}$, MPa	δ, %	ψ, %	N, cycles
	Hardening	Tempering, aging					
15Kh12N2MVFAB (0.15C, 0.5Si, 12Cr, 1.9Ni, 1.52Mo, 0.72W, 0.18V, 0.5Mn, 0.02N, 0.25Nb)	1393 K, 1 h	953 K, 2 h	$\frac{1080}{1060}$	$\frac{940}{950}$	$\frac{16}{4}$	$\frac{62}{10}$	$\frac{1029}{43}$
10Kh11N23T3MR (0.05C, 0.4Si, 11.4Cr, 23.2Ni, 1.48Mo, 2.98Ti, 0.62Al, 0.015B, 0.33Mn)	1373 K, 1 h	1000 K, 16 h + 923 K, 5 h	$\frac{1200}{1210}$	$\frac{880}{880}$	$\frac{29}{20}$	$\frac{47}{23}$	$\frac{3000}{1020}$
10Kh15N27T3MR (0.09C, 0.6Si, 15Cr, 27.11Ni, 1.41Mo, 1.92W, 2.85Ti, 0.29Al, 0.02B, 0.1Co)	1373 K, 1 h	1023 K, 16 h + 923 K, 10 h	$\frac{1270}{1240}$	$\frac{870}{880}$	$\frac{17}{10}$	$\frac{23}{10}$	$\frac{2277}{455}$
KhN43BMTYu (0.005C, 0.18Si, 0.3V, 14.5Cr, 44Ni, 1.8Mo, 1.67Ti, 0.5Al, 2.7Nb)	1373 K, 1 h	1023 K, 10 h + 923 K, 10 h	$\frac{1160}{960}$	$\frac{780}{740}$	$\frac{25}{4}$	$\frac{38}{14}$	$\frac{2560}{614}$
KhN55MBYu (0.05C, 0.23Si, 19.0Cr, 8.87Mo, 1.73Nb, 1.49Al, 12.0Fe, 0.02Cu, Ni – rest)	1323 K, 1 h	1000 K, 15 h + 923 K, 10 h	$\frac{1080}{970}$	$\frac{650}{660}$	$\frac{35}{5}$	$\frac{38}{19}$	$\frac{3200}{199}$

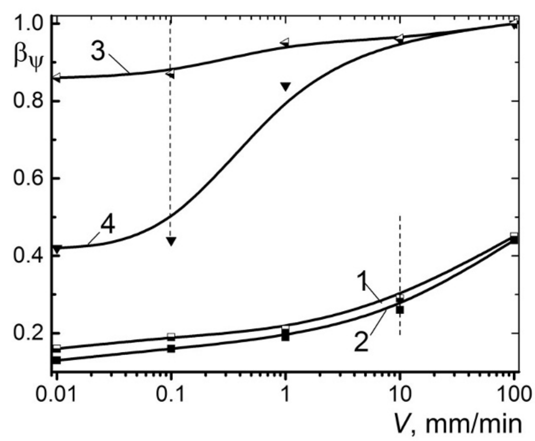

Fig. 1. Dependence of the coefficient of hydrogen influence on the lateral contraction β_ψ of specimens of 15Kh12N2MVFAB martensitic steel (*1*, *2*) and 10Kh15N27T3MR dispersion-hardening austenitic steel (*3*, *4*) on the extension rate: (*1*, *3*) hydrogen-unsaturated specimens, (*2*, *4*) specimens saturated with hydrogen at 623 K and a hydrogen pressure of 35 MPa for 10 h [15Kh12N2NVFAB (C_H = 5 ppm) and 10Kh15N27T3MR (C_H = 15 ppm)].

tension, the properties of carbon steels deteriorate only over a pressure range of 0–10 MPa, whereas the embrittlement of hardened specimens of Inconel 718 alloy begins at a pressure of 10 MPa and is enhanced until 70 MPa [3, 8, 12]. The effect of hydrogen on the ductility characteristics of steel and many other materials increases throughout the studied pressure range (0–70 MPa) [2, 8, 10–12].

In short-time static tension and low-cycle fatigue tests, two types of dependences of mechanical properties on hydrogen pressure have been revealed. One of them is typical of 15Kh12N2MVFAB martensitic steels (Fig. 2) and high-nickel alloys (over 55 wt.% Ni) (Fig. 3), when ductility and life decrease sharply at a low hydrogen pressure, and after reaching the pressure of 10 MPa (martensitic steels) or 60 MPa (KhN55MBYu alloy), the unfavorable action of hydrogen is stabilized. Another type of dependences is typical of dispersion-hardening austenitic chromium-nickel steels and alloys such as 10Kh15N27T3MR (curves *1* and *3* in Fig. 4) and

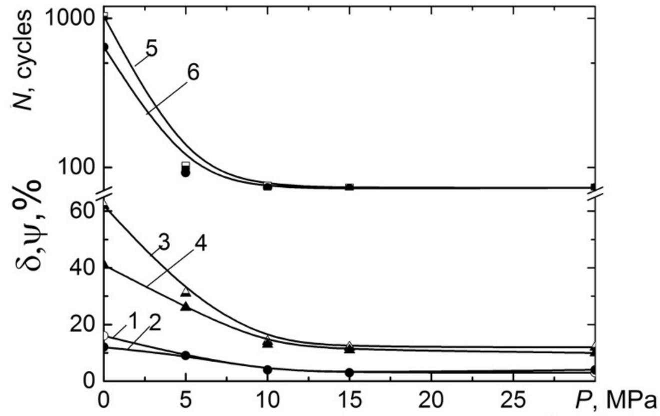

Fig. 2

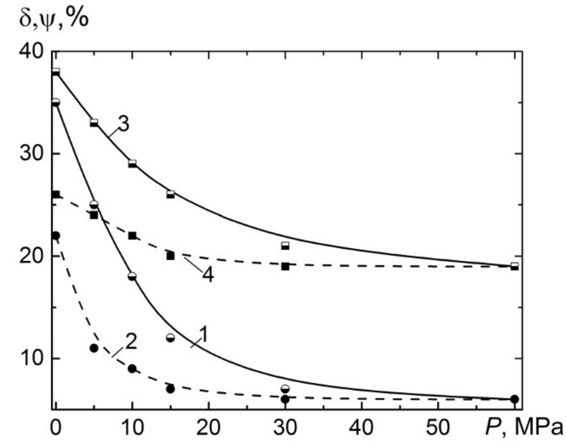

Fig. 3

Fig. 2. Dependence of the specific elongation δ (*1*, *2*), lateral contraction ratio ψ (*3*, *4*) ($V = 0.1$ mm/min) and the number of cycles to fracture N (*5*, *6*) ($\varepsilon = 1.6\%$) of specimens of 15Kh12N2MVFAB martensitic steel on the hydrogen pressure P at 293 K: (*1*, *3*, *5*) hydrogen-unsaturated specimens; (*2*, *4*, *6*) hydrogen-saturated specimens ($C_H = 5$ ppm).

Fig. 3. Dependence of the ductility characteristics δ (*1*, *2*) and ψ (*3*, *4*) of a KhN55MBYu nickel alloy on the hydrogen pressure P at a strain rate of 0.1 mm/min: (*1*, *3*) hydrogen-unsaturated specimens; (*2*, *4*) hydrogen-saturated specimens ($C_H = 24$ ppm).

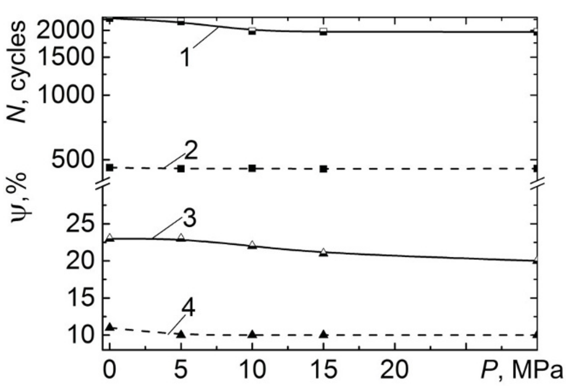

Fig. 4

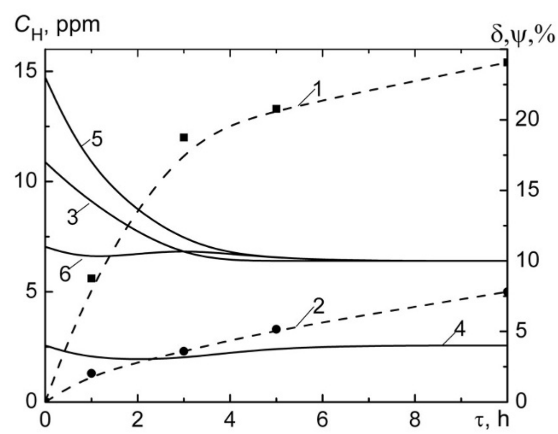

Fig. 5

Fig. 4. Dependence of the low-cycle life N ($\varepsilon = 1.6\%$) (*1*, *2*) and lateral contraction ratio ψ ($V = 0.1$ mm/min) (*3*, *4*) of specimens of 10Kh15N27T3MR austenitic steel on the hydrogen pressure P: (*1*, *3*) hydrogen-unsaturated specimens; (*2*, *4*) hydrogen-presaturated specimens ($C_H = 15$ ppm).

Fig. 5. Dependence of the absorbed hydrogen concentration C_H (*1*, *2*), specific elongation δ (*3*, *4*), and lateral contraction ratio ψ (*5*, *6*) of specimens of 15Kh12N2MVFAB (*2*, *4*, *6*) and 10Kh15N27T3MR (*1*, *3*, *5*) steels in hydrogen under a pressure of 35 MPa at $T = 293$ K ($V_{def} = 6.7 \cdot 10^{-5}$ s^{-1}) on hydrogen saturation time (623 K, 35 MPa, H_2).

KhN43BMTYu [10, 11, 18], whose properties deteriorate only slightly with increasing pressure and do not reach minimum values even at 60 MPa. In the absence of preabsorbed hydrogen, the ductility of 10Kh11N23T3MR steel does not deteriorate at all in a hydrogen atmosphere even at a pressure of 60 MPa and a rate of 0.01 mm/min [10].

In that case, to fully assess the sensitivity to hydrogen embrittlement, the specimens were presaturated with hydrogen, which simulated long operation of components in hydrogen. The additional effect of preabsorbed hydrogen is determining in the embrittlement of dispersion-hardening austenitic steels and alloys: 10Kh11N23T3MR, KhN43BMTYu [10, 11, 13], 10Kh15N27T3MR (Fig. 4) and significant for the KhN55MBYu alloy (Fig. 3). The influence of hydrogen on the mechanical properties of 10Kh11N23T3MR and 10Kh15N27T3MR steels increases with increase in its concentration to 10–12 ppm [10] and 13–14 ppm (Fig. 5), respectively; further increasing C_{H} does not cause additional reduction in the ductility of specimens.

Thus, at 293 K, the action of the external hydrogen atmosphere and preabsorbed internal hydrogen is determined by the structural class and the nickel content of the material. The degree of embrittlement of EK-62 and EP-901 nickel alloys (56 and 60 wt.% Ni) and heat-resistant 15Kh12N2MVFAB and EP-810 martensitic steels is determined by the gaseous hydrogen pressure, and the additional action of preabsorbed hydrogen is perceptible only at a low pressure (Fig. 2). The properties of 10Kh11N23T3MR and 10Kh15N27T3MR steels (23–28 wt.% Ni) deteriorate only after hydrogen saturation and change only slightly with increasing hydrogen atmosphere pressure (Fig. 4), and KhN43BMTYu and KhN55MBYu iron-nickel alloys are sensitive to the action of the external hydrogen environment and preabsorbed internal hydrogen (Fig. 3).

Thus, to evaluate the performance of martensitic steels and high-nickel alloys from low-cycle fatigue and short-time static tension characteristics, it is sufficient to carry out tests in gaseous hydrogen at a pressure of over 10 MPa, where as stable austenitic chromium-nickel steels and alloys with a nickel content of up to 55% must be presaturated with hydrogen [10, 11, 13]. Hydrogen absorbed at elevated temperatures has the strongest effect on the properties of the investigated dispersion-hardening iron-nickel steels and alloys at 293 K at its content of 15–30 ppm; embrittlement does not increase with further hydrogen saturation [10, 11, 13]. These critical values of hydrogen concentration are different in different materials are probably determined by their phase-structural composition, which depends on the percentage of nickel and carbides and intermetallics. For martensitic steels, the strain rate must not exceed 10 mm/min ($6.7 \cdot 10^{-3}$ s^{-1}) and for steels with an austenitic structure, 0.1 mm/min ($6.7 \cdot 10^{-5}$ s^{-1}). Under these conditions, a hydrogen embrittlement limit is reached: minimum values of performance characteristics (ductility, low-cycle life), which do not decrease with increasing adsorbed hydrogen pressure and absorbed hydrogen content.

Temperature Dependences of the Degree of Hydrogen Embrittlement of Iron-Nickel Alloys. The effect of temperature on the sensitivity to the action of hydrogen in static tension and low-cycle fatigue tests is qualitatively the same; there fore, the regularities obtained are illustrated for lateral contraction ratio as an example. The embrittlement of martensitic steels in gaseous hydrogen is greatest at 300–500 K (curve *1* in Fig. 6), of dispersion-hardening 10Kh15N27T3MR austenitic steel at 470–800 K (curve *2* in Fig. 6), and of KhN43BMTYu alloy at 370–970 K (curve *3* in Fig. 7). Thus, a shift of the range of embrittlement of austenitic steels with intermetallic hardening in hydrogen atmosphere to higher temperatures in comparison with martensitic steels has been established for the first time. The lateral contraction ratio ψ of 10Kh15N27T3MR decreases most greatly (by a factor of almost two) in hydrogen under a pressure of 35 MPa at 423 K and the low-cycle life N by a factor of five at 573 K. After the presaturation with hydrogen of austenite hardened with intermetallics (10Kh15N27T3MR steel and KhN43BMTYu alloy), the hydrogen embrittlement maximum shifts to room temperature (curve *3* in Fig. 6 and curve *4* in Fig. 7).

With increasing temperature, the strength of martensitic steel decreases sharply, and its sensitivity to the action of hydrogen decreases; at 623 K, its properties in hydrogen and helium are much the same (curve *1* in Fig. 6). More heat-resistant 10Kh15N27T3MR steel and KhN43BMTYu alloy retain high strength to the maximum temperature studied (1073 K), at which ductility and low-cycle life decrease in hydrogen by 20% (curves *2* and *3* in Fig. 6 and curves *3* and *4* in Fig. 7). The temperature dependences of the coefficients of influence of hydrogen on the characteristics of KhN55MBYu alloy are more complex. With increase in temperature from 773 to 973 K, the influence of hydrogen on the specific elongation, lateral contraction ratio and life of specimens decreases and increases at 1073 K (curves *1* and *2* in Fig. 7). The test results for dispersion-hardening austenitic steels and alloys [7, 10, 11, 13] indicate the existence of several types of their high-temperature behavior in hydrogen. One of them is characteristic of materials with a small percentage of refractory elements (10Kh11N23T3MR steel and KhN55MBYu alloy). The duration of extension at a rate of 0.1 mm/min at 1073 K in a neutral environment reaches 2 h, i.e., is

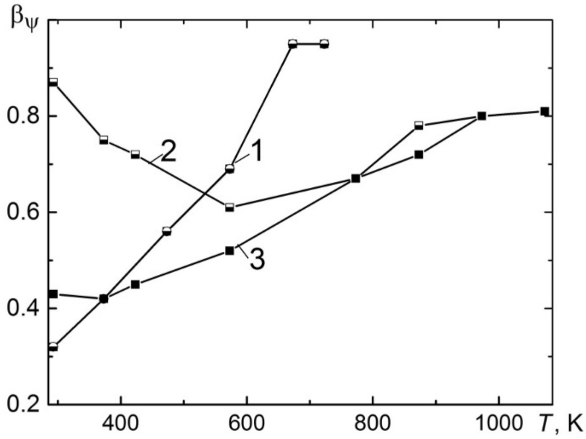

Fig. 6

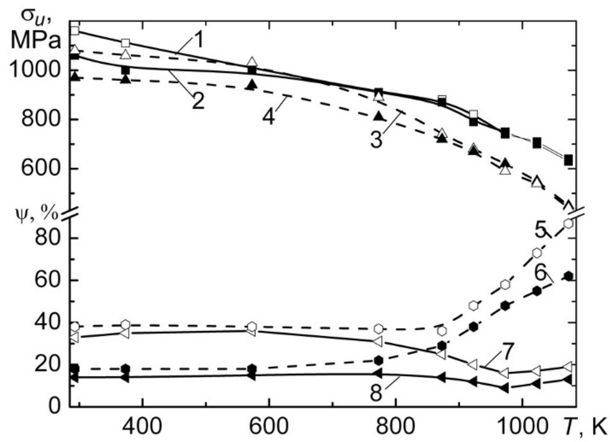

Fig. 7

Fig. 6. Temperature dependence of the coefficient of hydrogen influence on the lateral contraction ratio β_ψ ($V = 0.1$ mm/min) of 15Kh12N2MVFAB (*1*) and 10Kh15N27T3MR (*2*, *3*) steels at a hydrogen pressure of 35 MPa: (*1*, *2*) hydrogen-unsaturated specimens; (*3*) hydrogen-saturated specimens ($C_H = 15$ ppm).
Fig. 7. Temperature dependence of the coefficient of hydrogen influence on the lateral contraction ratio β_ψ ($V = 0.1$ mm/min) of Kh43BMTYu (*1*, *2*) and Kh55MBYu (*3*, *4*) alloys at a hydrogen pressure of 35 MPa: (*1*, *3*) hydrogen-unsaturated specimens; (*3*, *4*) hydrogen-saturated specimens (19 and 24 ppm, respectively).

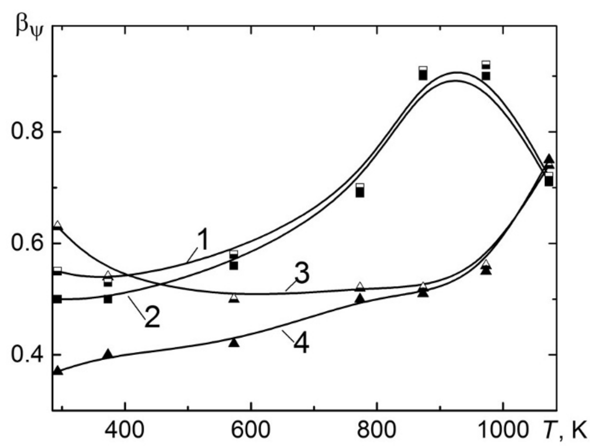

Fig. 8. Temperature dependence of the ultimate strength σ_u (*1–4*) and lateral contraction ratio ψ (*5–8*) of KhN43BMTYu (*1*, *2*, *7*, *8*) and KhN55MBYu (*3–6*) alloys in helium (*1*, *3*, *5*, *7*) and in hydrogen at a pressure of 35 MPa after hydrogen presaturation to a hydrogen content of 19 ppm (*2*, *4*, *6*, *8*) ($V = 0.1$ mm/min).

sufficient for the coagulation of intermetallics [18–22], which causes an intensive loss of strength and plasticization (curves *3–6* in Fig. 8). Through hydrogen-induced strain localization processes, the intergranular fracture of specimens at 1073 K occurs more rapidly, and there is no plasticization, which manifests itself by the occurrence of high-temperature hydrogen embrittlement of KhN55MBYu alloy (Fig. 7, curves *1* and *2*). Another type of behavior is typical of 10Kh15N27T3MR steel and KhN43BMTYu alloy with more Mo, Nb, W, whose strength decreases less with increasing temperature; there is no high-temperature plasticization at 1073 K (curves *1* and *2* in Fig. 8). Accordingly, the effect of hydrogen on their ductility decreases monotonically over a temperature range of 293–1073 K (curves *7* and *8* in Fig. 8), though remaining rather significant [10, 11, 13].

Thus, the existence of two (low- and high-temperature) extremes of the hydrogen embrittlement of heat-resistant austenitic steels and alloys with intermetallic hardening has been revealed in a temperature range of 293–1073 K. The low-temperature minimum of the mechanical properties of dispersion-hardening austenitic steels and alloys in hydrogen increases from 293–573 K (typical of martensitic and homogeneous austenitic steels) to 800–970 K. High-temperature hydrogen embrittlement manifests itself at 1073 K in steels and alloys with intermetallic hardening and a small percentage of refractory alloying elements (Mo, Nb, W), which retard phase transformations during tests.

CONCLUSIONS

1. The existence of a hydrogen degradation limit, the minimum value of the performance characteristics (ductility, low-cycle life), which do not decrease with increasing adsorbed hydrogen pressure and absorbed hydrogen content, has been established. Such limiting small values of the mechanical characteristics of martensite-aging steels and high-nickel alloys (> 55 wt.% Ni) are achieved at hydrogen pressures of over 10 and 30 MPa, respectively, and of stable dispersion-hardening austenitic steels and alloys (20–55 wt.% Ni) at a preabsorbed hydrogen content of 15–30 ppm.

2. The existence of two (low- and high-temperature) extremes of the hydrogen embrittlement of heat-resistant austenitic steels and alloys with intermetallic hardening has been reveled in a temperature range of 293–1073 K. The low-temperature minimum of the mechanical properties of dispersion-hardening austenitic steels and alloys in hydrogen increases from 293–573 K (typical of martensitic and homogeneous austenitic steels) to 800–970 K. High-temperature hydrogen embrittlement manifests itself at 1073 K in steels and alloys with intermetallic hardening and a small percentage of refractory alloying elements (Mo, Nb, W), which retard phase transformations during tests.

3. The obtained regularities of hydrogen degradation of martensitic and austenitic steels and nickel alloys will make it possible to choose structural materials and the safe operating conditions of components of gas turbines and power equipment that operate in hydrogen-containing gaseous environments.

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