

## PERSPECTIVE ALLOY OF THE Zr-Nb-Cr SYSTEM FOR THE NEW GENERATION OF NUCLEAR REACTORS

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Research of the Zr-Nb-Cr system alloys, promising for nuclear power engineering, with the aim of using them at operating temperatures of 500...600 °C for a new generation of nuclear reactors have been carried out. The possibility of increasing the high temperature strength, heat resistance, and cast-technological properties of the most promising serial Zr-1Nb alloy due to alloying it with 1 wt.% Cr. The improvement of properties is explained by the dispersion hardening of the alloy by the high-temperature, strong intermetallic ZrCr<sub>2</sub>. The high-temperature strength of the Zr-1Nb alloy can be increased by 30% in the temperature range 500...600 °C. The heat resistance of a promising alloy at a temperature of 500 °C exceeds the heat resistance of an industrial alloy Э635 by 30...50% and is at the level of heat resistance of the Э110 alloy at 20 hours exposure. The parabolic oxidation constants of the Zr-1Nb-1Cr alloy increase with increasing temperature and holding time, due to the formation of a dense scale of Cr<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub> oxides, instead of the brittle scale Nb<sub>2</sub>O<sub>5</sub>, ZrO<sub>2</sub>, which is characteristic for existing serial alloys Э110, E635, E125.

### INTRODUCTION

The creation of zirconium alloys with high-temperature strength and heat-corrosion resistance is of special interest in connection with the development of a new generation of nuclear reactors with superheated steam in which the shell temperature can reach 550 °C. Industrial alloys doped mainly with tin and niobium cannot be used in reactors with superheated steam because of low anticorrosive and mechanical properties at superheated steam temperature (> 500 °C).

The favorable combination of such characteristics as high melting temperature, heat resistance and corrosion resistance, low creep rate, low coefficient of thermal expansion and small capture cross section of fast neutrons make chromium and its alloys extremely promising for use as structural materials for the active zone of nuclear reactors on fast neutrons, thermonuclear reactors, as well as a number of units of nuclear power plants on thermal neutrons. In addition, chromium does not produce long-lived radioactive elements during irradiation.

Therefore, recently the main technical task in the development of materials for a new generation of nuclear reactors with superheated steam is to investigate the possibility of increasing the high-temperature strength and heat-corrosion resistance of zirconium alloys while maintaining manufacturability at the level of serial zirconium-niobium alloys due to doping of zirconium with chrome.

The purpose of this work was to study the change in heat resistance at temperatures of 500...600 °C, some strength characteristics and features of the casting-technological properties of the industrial zirconium alloy Э110 with its additional doping with an optimal amount of chromium. In the formulation of this work, it was also the task to investigate the possibility of formation in the Zr-Nb-Cr system of an alloy with a stable  $\alpha$ -phase, which is strengthened by intermetallics at eutectoid transformation of the  $\beta$ -phase.

### MATERIALS AND METHODS OF RESEARCH

Melting of the high-purity alloys of the Zr-Nb-Cr system were carried out in a argon-arc installation with a non-consumable tungsten electrode in copper crucibles with a diameter of 50 mm. Since the volatility of chromium vapor at the melting point is about 10<sup>-1</sup> MPa, smelting of highly pure alloys doped with chromium was carried out in an inert medium, argon at an argon overpressure of 0.02...0.04 MPa. To purify the argon, a preliminary re-melting of the hetero-Ti-Zr was carried out, which absorbs oxygen, nitrogen, water vapor and carbon compounds from the atmosphere of the furnace. The Zr-Nb-Cr system alloys were melted from iodide zirconium, electrolytic chromium and high purity niobium brand of NB-1.

The melted experimental samples of the alloys crystallized in a metal form (spherical and elongated oval), as well as in a copper mold of a special one-piece construction with casting inclinations and a special casting profitable part that provided feeding to the shrinkage defects of the casting during crystallization.

Investigation of the chemical composition of the alloys was carried out by the method of emission spectral analysis using the average dispersion apparatus of the ISP 30, an error of < 5%.

The homogeneity of the chemical composition of the castings was determined on a Camebax SX-50 electron probe X-ray microanalyzer with a probe from 3 to 35  $\mu\text{m}^2$ . The investigations were carried out as an integral probe with a probe 35x35  $\mu\text{m}$  with a spacing of 98  $\mu\text{m}$ , and zonal along the grain and grain boundaries with a probe of 1.5...3  $\mu\text{m}^2$ .

The content of O, N, H was studied by the method of reductive extraction in a helium stream-gas chromatography, on a VE-GB unit. The study of the content of C was carried out by coulometric titration at the pH value, on the Express Carbon Analyzer of grade AN-7529.

Metallographic studies of cast, deformed and after thermal treatments of alloys were carried out using a

light microscope MIM-7. The microhardness was measured on a PMT-3 device at a load of 100 g.

Heat treatment of alloys was carried out in vacuum  $10^{-3}$  Pa. The deformation (by draft) of the alloys was carried out on a hydraulic press of 50 tons at a speed of 2.5 mm/s.

Mechanical tests were carried out with a strain rate of  $1.1 \cdot 10^{-3} \text{ s}^{-1}$  for tension and compression in vacuum on a machine for mechanical tests of type 1246 P2 / 2300.

## RESULTS AND DISCUSSIONS

Chromium is a promising alloying element for the creation of structural zirconium alloys for nuclear power due to the small crossing of the capture of thermal neutrons, the rapid decay of induced activity [2], high heat resistance [3], the existence in the Zr-Cr system of a high-temperature [4], durable [5] intermetallide  $\text{ZrCr}_2$  and a relatively high temperature of the eutectoid transformation  $\beta \rightarrow \alpha + \text{ZrCr}_2$ . Taking into account the low solubility of chromium in zirconium, it is possible to obtain a high volume fraction of the  $\text{ZrCr}_2$  intermetallide stable to high temperatures. Doping with chromium provides many opportunities for strengthening zirconium [6, 7].

Studies were made of the possibility of increasing high-temperature strength and heat resistance of the  $\text{Э110}$  alloy with optimal chromium alloying, with the aim of using it at higher operating temperatures in a new generation of nuclear reactors with superheated steam in which the shell temperature can reach  $550 \text{ }^\circ\text{C}$ .

The prerequisite for this work was the results of our preliminary studies, which showed the possibility of increasing the heat resistance due to dispersion hardening in  $\text{Zr-(0.5...4.5)\%Cr}$  alloys while maintaining technological ductility and relatively high heat-corrosion resistance [1].

However, it should be noted that when chromium is introduced into zirconium alloys, the crystallization interval substantially increases, which significantly worsens the casting-technological characteristics of the alloys. This is the reason for the increased propensity of these alloys to form shells, shrinkage porosity, and also crystallization cracks and requires the creation of special casting systems. It is necessary to use special chill molds (with large slopes) or compliant with burning impurities of ceramic molding equipment. Directional feeding of the casting in the process of crystallization and vibration is required. However, an increase in the purity of alloys in implementation impurities improves the casting characteristics and reduces the number of crystallization and thermal cracks.

The most optimal content of chromium in zirconium alloys of Zr-Cr systems can be considered as the eutectoid composition  $\text{Zr-1.0\%Cr}$ , which has a relatively small crystallization interval for these alloys –  $190 \text{ }^\circ\text{C}$ , therefore, in order to increase the heat resistance and corrosion resistance of the industrial zirconium alloy  $\text{Э110}$  at temperatures on the order of  $500 \text{ }^\circ\text{C}$ , additional doping with  $1.0\% \text{ Cr}$  was tested.

Considering that niobium enters in the  $\text{Э110}$  high-tech serial alloy with narrow crystallization interval, and contributes to the reduction of the crystallization

interval of zirconium alloys, it is of interest to study the improvement in the properties of the  $\text{Zr-1Nb}$  and  $\text{Zr-1Cr}$  alloys in the  $\text{Zr-1Nb-1Cr}$  alloy.

In the complex alloying of zirconium alloys with chromium and niobium, hardening is achieved due to the following factors: dispersive hardening by Laves phases, solid solution particles based on niobium, solid-solution hardening and grain refinement.

Moreover, according to the data of [8], the joint introduction of Nb and Cr into the zirconium alloys in the optimum ratio leads to an increase in the heat resistance in air at  $650 \text{ }^\circ\text{C}$  for 20 hours to  $18.52 \text{ g/m}^2$  (at the level of pure zirconium –  $22 \text{ g/m}^2$ ).

Experimental studies of the most promising scientifically – determined alloys of the  $\text{Zr-1\% Nb-(0.5...1.5)\%Cr}$  system have been carried out.

When choosing promising alloys, microtests, heat resistance, microstructure and manufacturability in the cast state were investigated.

Since the oxidation of zirconium in superheated water or water vapor is very similar to oxidation in a gaseous medium ( $\text{O}_2$ , air,  $\text{CO}_2$ ), we conducted a study of the effect of chromium on the heat resistance of zirconium in air.

In the study of new experimental alloys, parallel studies were carried out on the serial  $\text{Э110}$ ,  $\text{Э635}$ ,  $\text{Э125}$ , cast alloys according to the same technology as the experimental alloys (Tabl. 1).

Table 1  
Comparative microhardness and heat resistance of experimental cast alloys of Zr-Nb-Cr system

Alloys	Comparative microhardness and heat resistance alloys		
	Micro hardness, MPa	Heat resistance at $500 \text{ }^\circ\text{C}$ , $\text{mg/cm}^2$	
		Heating time, hour	
		10	50
Zr-1Nb ( $\text{Э110}$ )	2200	0.53	0.88
Zr-1Nb-1Cr	2700	0.55	0.90
Zr-1Nb-0.5Cr-0.5Fe	1800	1.09	3.94
Zr-1Nb-1Cr-1Fe	1990	0.95	2.34
Zr-1Nb-0.35Fe-1.2Sn ( $\text{Э635}$ )	1460	0.81	2.12
Zr-2.5Nb ( $\text{Э125}$ )	1640	1.06	2.2
Zr-2.5Nb-0.5Cr-0.5Fe	2140	1.51	7.2

Studies have shown that the microhardness of the alloys of the Zr-Nb-Cr system exceeds the microhardness of the  $\text{Э635}$  alloy by  $30...50\%$ . This indicates the effectiveness of dispersed hardening of alloys with chromium intermetallides. The highest microhardness among the experimental alloys in the cast state has the alloy  $\text{Zr-1Nb-1Cr}$ .

The possibility of increasing the strength characteristics of the  $\text{Э110}$  alloy with its optimal doping with chromium is indicated by an increase in

microhardness of  $\sim 1.3$  times in different structural states of the alloy (Tabl. 2).

Table 2  
Microhardness cast alloys  $\text{Э110}$  and  $\text{Э110}$ , alloying with chromium

Alloys/ state	Microhardness, MPa		
	Iodide	Cast	Annealing at 600 °C, 4 hours
Zr	1060... 1150	–	–
$\text{Э110}$	–	2300... 2360	2100... 2190
$\text{Э110+}$ 1% Cr	–	2900... 2970	2700... 2800

The increase in the microhardness of the alloy  $\text{Э110}$  as a result of doping it with chromium, which remains after annealing, also indicates the thermal stability of the dispersed hardening of alloys with chromium intermetallides.

It is known that one of the factors that determine the quality of cast alloys is their structure.

Since the quality of the cast ingots determines the quality of the alloys after the thermoplastic processing, when selecting the composition of prospective high-temperature zirconium alloys, great importance is attached to the features of the formation of the cast billets structure. Due to the control of both the dispersion of the cast structure and its morphology, it is possible to simultaneously increase both strength characteristics and plasticity.

As shown by our previous studies [9], the thermophysical characteristics of low-alloy zirconium alloys are comparatively low thermal conductivity (the thermal conductivity coefficient is  $c = 25...31 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ ) and the heat capacity ( $C_p = 0.27...0.38 \text{ kJ}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$ ) reduce the dependence of the crystallization of these alloys on the supercooling conditions, which is characteristic of a number of refractory metals. Therefore, it is possible to improve the quality of low-alloy cast zirconium alloys in the structural plan either by cleaning the grain boundaries, or by creating a fine-grained “basket” morphology of the  $\alpha$ -phase. In cast alloys this, primarily, depends on the composition of the alloy, as well as on the technological parameters of the smelting – the degree of refining and the uniformity of the alloying components distribution.

Comparative metallographic studies of the typical morphology of the output cast structures of promising experimental alloys of the Zr-Cr system and the serial alloys  $\text{Э110}$  and  $\text{Э635}$  showed that increasing both the strength and the characteristics of the heat resistance of optimally alloyed alloys in the cast state contributes to the formation of a “basket” structure of the  $\alpha$ -phase hardened with drip dispersed chromium intermetallides at eutectoid transformation of the  $\beta$ -phase.

Metallographic studies of the most perspective alloy Zr-1Nb-1Cr confirmed that the formation “basket” structure of the  $\alpha$ -phase, which is strengthened by finely dispersed chromium intermetallides at the eutectoid transformation of the  $\beta$ -phase, also contributes to the increase of its strength characteristics (Fig. 1).

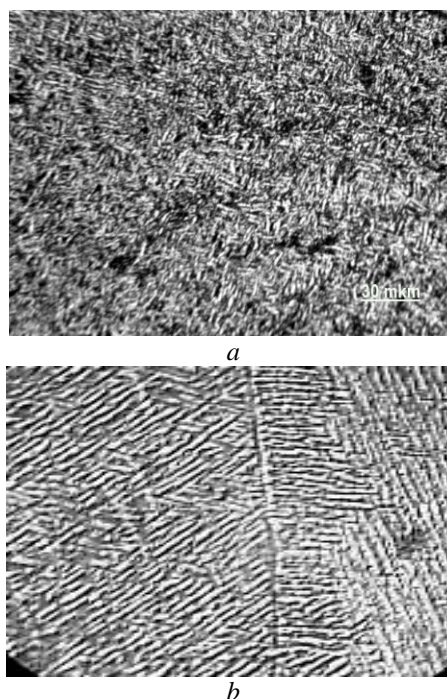


Fig. 1. Microstructure of cast alloys,  $\times 500$ :  
*a* –  $\text{Э110} + 1\% \text{ Cr}$ ; *b* –  $\text{Э110}$

The heat resistance of the most prospective Zr-1Nb-1Cr alloy at 500...600 °C is at the level of the  $\text{Э110}$  alloy and exceeds the heat resistance of the industrial alloy  $\text{Э635}$  by 30...50%. Moreover, near-surface loose film scale from mixtures of  $\text{Nb}_2\text{O}_5$  and  $\text{ZrO}_2$  oxides, which is typical for high-temperature ( $> 500$  °C) oxidation for  $\text{Э110}$  and  $\text{Э635}$  alloys, is absent in this alloy even at 600 °C (Fig. 2).

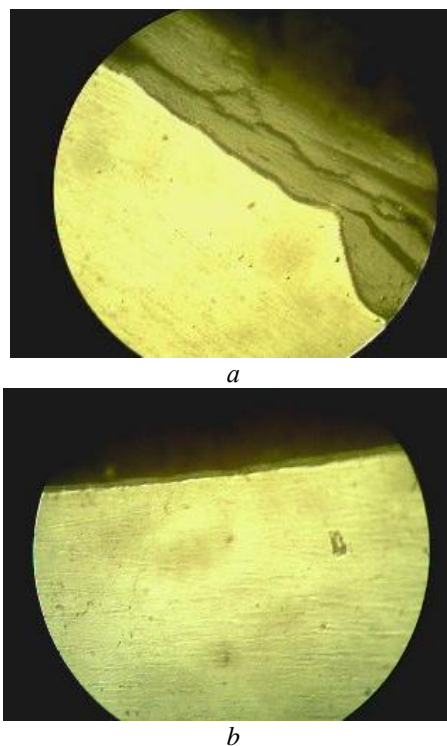


Fig. 2. A typical morphology of the structure the near-surface scale film of zirconium alloys samples after high-temperature (500 °C) oxidation:  
*a* – 500/50,  $\text{Э110}$ , cast; *b* – 500/50, Zr-1Nb-1Cr, cast

From the analysis of the structure of the near-surface film scale of Zr-Nb-Cr alloy samples after oxidation it has been established that alloy of this system with chromium content, which corresponds to the eutectoid composition of the Zr-Cr alloy, have minimal oxidation.

Moreover, metallographic studies of scale have shown that in the perspective alloy Zr-1Nb-1Cr at a temperature of 500 °C for 10...50 hours, a dense scale of 10...15 μm is formed from the oxides of Cr<sub>2</sub>O<sub>3</sub> and ZrO<sub>2</sub>, and in the existing serial alloys, Э110 (Э635) is brittle scale from 100 to 140 μm from oxides of Nb<sub>2</sub>O<sub>5</sub>, ZrO<sub>2</sub>, which indicates a higher parabolic oxidation constant at increase of temperatures and the heating up time in existing alloys than in the prospective alloy Zr-1Nb-1Cr.

In order to establish the cast-technological properties of the alloys, the temperature interval of crystallization of the most prospective alloys of the Zr-Nb-Cr system were investigated, since the the temperature interval of crystallization is the determining characteristic of the alloy technological and is the reason for the increased defectiveness of cast billets.

By the method of differential – thermal analysis on the installation VDTA-8M3 were investigated the temperature intervals crystallization and phase transformation intervals of prospective zirconium alloys Zr-Nb-Cr system (Zr-1Nb-1Cr-1Fe and Zr-1Nb-1Cr) as well as a serial process – zirconium-niobium alloy Zr-1Nb (Tabl. 3).

When performing differential - thermal analysis, the samples were heated in crucibles with Y<sub>2</sub>O<sub>3</sub> at a rate of 80 degrees/min, in an inert gas – helium. The recording was carried out on a two-coordinate sampler PDP-4002.

The thermograms of the investigated alloys confirmed that the introduction the optimal amount of chromium into the alloy Э110 does not exceed the temperature intervals of crystallization of alloys above 80...90°, that is quite reasonable to cast alloys. In this case, the casting-technological properties of Zr-1Nb-1Cr alloy rather altitude and are at the level of the serial alloy Э110. Thus, the alloy Zr-1Nb-1Cr, the crystallization interval is 90°, has a free casting shrinkage 1...1.5%, volume of 3...4%; micro cracks and intercrystal porosity in the samples are not recorded.

Table 3  
Intervals of crystallization temperatures and phase transformation temperatures of prospective experimental alloys of the Zr-Nb-Cr system

Alloys	Intervals of crystallization temperatures and phase transformation temperatures	
	Intervals of crystallization temperatures (degree)	Phase transformation temperatures (degree)
Zr-1Nb	1850...1800	850...620
Zr-1Nb-1Cr	1865...1770	867...765
Zr-1Nb-1Cr-1Fe	1855...1740	810...747

Taking into account, that the temperature of the α→β-phase transition in the alloy is a characteristic of the maximum operating temperature of the alloy, it follows that the Zr-1Nb-1Cr alloy can retain the increased heat-resistance characteristics up to 650...700 °C, that is, 150...200 °C higher than the Э110 serial alloy.

## CONCLUSIONS

1. Researches on development of an alloy of Zr-Nb-Cr system with increased high-temperature strength and heat – resistance due to optimal chromium alloying of Zr-1Nb alloy for the purpose of possibility of its use at operating temperatures of 500...600 °C.

2. Studies have shown that introduction of chromium in an amount of 0.9...1.1% in the Zr-1Nb alloy (composition approximate to the eutectoid for the Zr-Cr alloys system) gave the new alloy properties that are not owned by the known zirconium alloys, namely:

- higher characteristics of strength (due to strengthen αZr-phase an alloy by the uniformly distributed fine dispersivity intermetallic particles of ZrCr<sub>2</sub>) at saving of sufficient plasticity (due to optimal alloys composition close to eutectoid for the Zr-Cr system, the ratio of dispersible cast structure and its morphology – “basket” structure);

- increased heat-corrosion resistance at 500 °C due to changes in the kinetics of oxidation and the formation of a dense scale of Cr<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub> oxides in the near-surface film scale instead of loose scale from oxides of Nb<sub>2</sub>O<sub>5</sub>, ZrO<sub>2</sub> in serial alloys (Э110, Э635, Э125);

- increased the cast-technological properties of the alloys (due to a relatively narrow interval of crystallization and phase transformation of the alloy, as well as due to the dispersity and “basket” microstructure of the alloy).

3. Using of the proposed technical solution:

- opens the possibility of using the alloy in a new generation of nuclear reactors, in which the shell opens the possibility of using the alloy to create a new generation of nuclear reactors, in which the reactor shell temperature can reach 550 °C;

- allows significantly reduce the financial costs when using the proposed alloy in existing nuclear reactors by increasing the life of a more heat-corrosion-resistant alloy;

- allows its use as a konstruktural material for creating heat-resistant products (operating temperature up to 600 °C) in chemical engineering, shipbuilding and others.

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## **ПЕРСПЕКТИВНЫЙ СПЛАВ СИСТЕМЫ $Zr-Nb-Cr$ ДЛЯ НОВОГО ПОКОЛЕНИЯ ЯДЕРНЫХ РЕАКТОРОВ**

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Проведены исследования сплавов системы  $Zr-Nb-Cr$ , перспективных для ядерной энергетики, с целью возможности использования их при рабочих температурах 500...600 °С для атомных реакторов нового поколения. Установлена возможность повышения жаропрочности, жаростойкости и технологичности наиболее перспективного серийного сплава  $Zr-1Nb$  за счет легирования его 1 мас.%  $Cr$ . Улучшение свойств объясняется дисперсным упрочнением сплава высокотемпературным, прочным интерметаллидом  $ZrCr_2$ . При этом жаропрочность сплава  $Zr-1Nb$  может быть повышена на 30% в температурном интервале 500...600 °С. Жаростойкость перспективного сплава при температуре 500 °С превышает жаростойкость промышленного сплава Э635 на 30...50% и находится на уровне жаростойкости сплава Э110 при выдержке 20 ч. Константы параболического окисления сплава  $Zr-1Nb-1Cr$  с повышением температуры и времени выдержки уменьшаются в результате образования плотной окалины из оксидов  $Cr_2O_3$ ,  $ZrO_2$  вместо хрупкой окалины  $Nb_2O_5$ ,  $ZrO_2$ , характерной для существующих серийных сплавов Э110, Э635, Э125.

## **ПЕРСПЕКТИВНИЙ СПЛАВ СИСТЕМИ $Zr-Nb-Cr$ ДЛЯ НОВОГО ПОКОЛІННЯ ЯДЕРНИХ РЕАКТОРІВ**

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Проведено дослідження сплавів системи  $Zr-Nb-Cr$ , перспективних для ядерної енергетики з метою можливості використання їх при робочих температурах 500...600 °С для атомних реакторів нового покоління. Встановлено можливість підвищення жароміцності, жаростійкості і технологічності найбільш перспективного серийного сплаву  $Zr-1Nb$  за рахунок легування його 1 мас.%  $Cr$ . Поліпшення властивостей пояснюється дисперсним зміцненням сплаву високотемпературним, міцним інтерметалідом  $ZrCr_2$ . При цьому жароміцність сплаву  $Zr-1Nb$  може бути підвищена на 30% в температурному інтервалі 500...600 °С. Жаростійкість перспективного сплаву при температурі 500 °С перевищує жаростійкість промислового сплаву Э635 на 30...50% і знаходиться на рівні жаростійкості сплаву Э110 при витримці 20 годин. Константи параболического окиснення сплаву  $Zr-1Nb-1Cr$  з підвищенням температури і часу витримки зменшуються в результаті утворення щільної окалини із оксидів  $Cr_2O_3$ ,  $ZrO_2$  замість крихкої окалини  $Nb_2O_5$ ,  $ZrO_2$ , характерної для існуючих серийних сплавів Э110, Э635, Э125.