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STEELS FOR WORK IN SUPERCRITICAL WATER

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Supercritical water (SCW) contains traces of chlorides, oxygen, and hydrogen, which forms during the thermal decomposition of water. You can't rule out the presence of peroxide compounds, which can form even without radiation, judging by the oxidizing ability of SCW and without adding oxidizing agents. So, the set of factors that create a corrosive environment in SCW, which could be used for reactor cooling, is quite large, and the requirements for materials working in SCW are high.

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INTRODUCTION

Since it is assumed that steels should operate in nuclear power plants in a water environment at temperatures no lower than 370^0 and a corresponding pressure of 22 MPa, they should have higher requirements than steel 08X18H10T or steels of the 300 series (according to American classification), which are currently used in reactor construction. Alloying has a complex effect on the quality of the alloy. Chromium increases hardness and corrosion resistance, nickel increases plasticity, molybdenum prevents red-shortness and oxidation at high temperatures, manganese gives impact strength and wear resistance, titanium provides elasticity and helps remove oxygen during smelting, silicon gives corrosion resistance and stability, nitrogen strengthens, and aluminum protects steel at high temperatures by forming an Al_2O_3 film. Some impurities are incompatible, like titanium and nitrogen. The most interesting steels for use in SCW might be AL-6XN and Alloy 800, as well as the Haynes 214 alloy.

1. PROMISING STEELS (ALLOYS) FOR USE IN SCW-ENVIRONMENTS

AL-6XN Steel. AL-6XN steel (or alloy) is designated as No. 8367 in the Unified Numbering System (UNS), in Germany it is designated as W. Nr. 1.4529, its density is 8.055-8.06, it is a low-carbon, super-austenitic alloy, better than duplex stainless steels. The difference in chemical composition compared to "food-grade" stainless steel, which most nuclear power plant equipment is made from, is as follows: a slight difference in the amount of chromium, twice as much nickel, the presence of several percent of molybdenum, and up to a quarter percent of nitrogen, which determines the absence of titanium.

It can be expected that the price of this steel is significantly higher than 'food-grade' steel due to the presence of molybdenum and a higher amount of nickel. Steel products are used in cellulose and paper production, the construction of offshore oil and gas platforms, food processing, biopharma, combustion chambers, transformer cases, that come into contact with seawater, oil cracking facilities, other chemical equipment, and thermocouple cases. AL-6XN steel is made into strips, foil, wire, rods, bars, flanges, valves, fittings, pipes, and forgings.

It shows excellent resistance to intergranular corrosion and cracking, and great weldability. This alloy is used instead of the 300 series stainless steels at high temperatures in acidic environments. Compared to 08X18H10T steel, this grade has higher ductility (formability) and corrosion resistance [1]. In terms of its properties, steel (alloy) AL-6XN outperforms ordinary steels and comes close to nickel alloys, such as the 08X18H10T alloy [2], that is it is a super steel. The AL-6XN alloy is very heat-resistant, with the temperature at which scale formation intensifies 1100 °C.

The steel is ductile, with an elongation at break of 30…35% (or more). The melting temperature range is 1320...1400° [3]. AL-6XN steel (Cr20Ni24Mo6N) with nitrogen as a strengthening alloying element outperforms 304L and 316L type steels at elevated temperatures (600 °C). Even better properties are seen in modified steel, that is made by adding small amounts of zirconium and tantalum [4]. The domestic counterpart of AL-6XN steel, known as XH32T, is very closed to it's properties. The difference is that the domestic version does not contain the nitrogen strengthening element. This alloy is used for making high-temperature equipment in petrochemical production, including ammonia production, nuclear reactor steam generators, and heating element cases. The chemical composition of the two steels is practically identical. Therefore, research data on the corrosion of domestic steel can also be applied to the American one. The chemical composition of the steel, compared to so-called 'food-grade' steel, is shown in Table 1.

Alloy 800 steel. The alloy or steel INCOLOY Alloy 800 is also a superaustenitic stainless steel, with iron content not exceeding 50%, chromium at 20%, nickel at 30%, and titanium and aluminum at 0.5% each.

Alloy 800 steel differs from AL-6XN steel by the presence of aluminum and titanium, the absence of nitrogen and molybdenum, and a higher nickel content. Trade name synonyms include INCOLOY 800, Alloy 800, UNS No8800, Werkstoff Nr.1.4876, Ferrochronin 800, Nickelvac 800, Nicrofer 3220, Nicrimphy 800, Pyromet 800.

Table 1

Composition of steels AL-6XN and AISI-304, weight %

Elements	AL-6XN sample from Canada	AL-6XN [5]	AISI-304 [5] (S30400)
C	0.02	0.03	0.08
Cr	20.62	20…22.0	8.0…10.5
Cu	0.31	0.75	–
Fe	Bal	–	–
Mn	0.42	–	2.0
Mo	6.16	6.0...7.0	–
N	0.2	0.18	–
Ni	23.81	23.5…25.5	8.0…10.5
P	0.024	–	–
S	0.0001	–	–
Si	0.42	–	–

Table 2

Chemical composition of steels Alloy 800 and 08X18H10T, weight %

Elements	Alloy 800 Sample from Canada	Alloy 800 by [6]	Alloy 800 by [7]	Alloy 800 by [8]	08X18H10T by our information
Al	0.51	0.15…0.60	0.52	–	–
C	0.07	0.10	0.08	–	0.06
Co	0.04	2.00	–	–	–
Cr	20.62	19…23.00	19.71	20.40	17.93
Cu	0.04	0.75	0.25		0.17
Fe	46.7	bal	45.6	49.88	–
Mn	0.59	1.50	0.73	0.92	1.07
Mo	–	–	–	–	0.08
N	0.016	–	–	–	–
Ni	30.36	30…35.00	31.52	27.64	9.89
P	0.014	–	0.017	0.024	0.019
S	0.0002	–	0.001	0.010	0.033
Si	0.3	0.015	0.32	–	0.05
Ti	0.55	0.15…0.60	0.58	0.44	0.62
V	–	–	–	–	0.04
W	–	–	–	–	0.05

The steel composition is shown in Table 2, where the second column lists the data from Canadian researchers who provided us with steel samples, the third column has the manufacturer's data, the fourth column shows data from [7], the fifth from [8], and the sixth shows, for comparison, the data for steel 08X18H10T obtained by us using the Spectrolab M10 emission spectrometer (Germany). The operating temperature range for Alloy 800 steel is -200 to 815°. This steel is widely used for manufacturing equipment that requires corrosion resistance, heat resistance, high strength, and stability when operating at temperatures up to 800°. The alloy is resistant in many water environments and, due to it's high nickel content, it resists stress corrosion cracking. At elevated temperatures, it is resistant to oxidation, carburization, sulfidation, as well as tensile stress and creep. For use at temperatures above 800 °C, INCOLOY 800H and 800HT alloys are used, which have the same chemical composition as Alloy 800 but differ in grain size. Alloy 800 steel is used to make baskets and trays for heat treating various parts. It is suitable for making heater covers, retorts, and furnaces in the chemical industry and is also used at nuclear power plants. The steel has a density of 7.94 g/cm³ and a melting range of 1357…1385° [9].

Haynes 214 alloy. This alloy exceeds even super steels in heat resistance. Other names for Haynes 214 include UNS No7214, AWS 061, W. Nr 2.4646, Cabot 214, and Hastelloy 214. It outperforms most alloys in oxidation resistance at high temperatures and is mainly intended for use at 955 °C (1750 °F) and above. It's used to make ceramic firing trays, baskets, and furnace parts. Nickel is the base of this alloy. It contains up to 6% iron, up to 17% chromium, up to 5% aluminum, up to 2% cobalt, up to 1% tungsten, manganese, and molybdenum (each), zirconium up to 0.2%, and some samples have traces of yttrium and niobium up to 0.15% [10, 11]. The

alloy density is 8.05, and the melting temperature range is 1355…1400°.

2. STEEL CORROSION

According to Guzonas [12], SCW contains traces of chlorides, oxygen, and hydrogen, which forms during the thermodecomposition of water. The presence of peroxide compounds cannot be ruled out, as they can form even without radiation, judging by the oxidizing ability of SCW and without adding oxidizing agents. So, the range of factors that create a corrosive environment in supercritical water is quite wide, and radiation just makes the corrosion worse. In the work [13], different samples of Alloy 800 steel were studied at temperatures of 500…760 °C, and it was found that aluminum and titanium increase creep resistance. The steel samples had slightly different contents of aluminum and titanium. When studying the behavior of steel in nuclear corrosion tests at 400° with the addition of neutral salts KCl or NaCl, it was found that pitting (or spot) corrosion occurs [14]. Additionally, it was observed that the inner layer of corrosive deposits is chromium oxide, while the outer layer is a spinel of iron and nickel oxides. Currently, science and technology are working on implementing fission reactors with an operating temperature around 700…750 °C and helium gas cooling.

An attempt was made to assess the behavior of Alloy 800 steel in an inert helium atmosphere, but it turned out that the atmosphere was not completely inert [15]. Although high-purity helium (99.996%, purity grade 4.6) was used, during contact with the steel at temperatures of 700…750 °C over hundreds of hours, the helium accumulated gases such as H_2, H_2O, CO, CO_2, and CH_4 at levels of a few ppm, and even nitrogen up to 25 ppm, which led to further reactions with the metal. While the steel is at the above working temperature, processes of carburization and decarburization occur within it, along with diffusion of some alloy components to the grain surfaces and even to the surface of the steel sample, where they form oxide films of Cr and Mn of unstable composition. These processes become noticeable when steel samples are kept in a helium atmosphere for 150 to 1000 h. Aluminum, oxidizing earlier than other elements, forms oxide films that slow down the diffusion of elements both to the surface of the steel sample and to the grain surfaces, which preserves the outstanding properties of the steel at high temperatures. Alloy 800 steel is used as a material for steam generator tubes and other pipelines at nuclear power plants [8]. Before installation at the plants, the pipes underwent shot peening, etching, passivation, and electropolishing. Some types of surface treatment change the steel's structure and promote chromium diffusion to form an inner protective film layer. After being held for 72 h in an autoclave with water at 400 °C and 10 MPa pressure, the four steel samples showed, visually, the densest oxide films on the untreated steel sample and the one after shot peening, while a semi-transparent film formed on the polished surface. Three samples showed a mass gain, while the fourth – after shot peening – lost weight, likely due to the high surface roughness causing some of the formed oxides to flake off. A big drawback of the article is that the steel analysis doesn't include data on aluminum content, even though it's really important since Alloy 800 steel differs from other steels specifically in terms of aluminum, and its effect on chromium diffusion isn't considered, so the data obtained in the work are approximate.

According to American ASTM G-48B standards, AL-6XN steel showed pretty good resistance in a 10% ferric chloride solution. Crevice corrosion only started to appear at 43^0. This steel was introduced into production a little before 1970 and showed good resistance in salty and seawater with high chloride content. During 18 years of operation of thin-walled tubes in seawater, only minor pitting corrosion was observed. In studies of pitting and crevice corrosion of AL-6XN steel in solutions simulating seawater, the order of factor influence was established as follows: temperature, pH, chloride ion concentration. Many authors paid a lot of attention to modeling, but in our opinion, it would have been better to conduct real-life studies [16].

In the work [17], the possibility of pitting corrosion of AL-6XN steel in 60% acetic acid with impurities of ions and formic acid was practically studied. The threshold chloride concentration for corrosion of AL-6XN steel is 9 mg/l, whereas for 316 L and 2205 steels it is 3 and 5 mg/l respectively, which means that the steel grade we are interested in has an advantage over the other common steel grades.

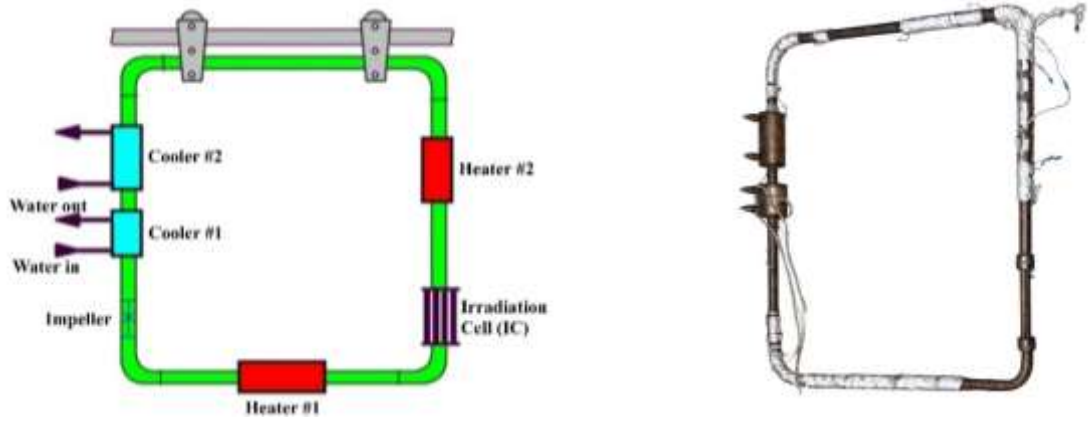
An original method for increasing the hardness and reducing the wear rate of the Haynes 214 alloy was proposed in study [18]. For this, the alloy was held at a temperature of -196 °C. The properties of the Haynes 214 alloy, compared with other alloys and steels, were examined in detail in study [19]; it is assumed that the alloy can operate for a long time at 760 °C and a pressure of 32 MPa. In study [20], to improve the corrosion resistance of the alloy at 800°, laser spraying of aluminum and chromium oxides was used. The composition of the tested samples was as follows (weight %) – Ni 75 (Balance), Cr 16, Al 4.5, Fe 3, Y 0.01. Chromium oxide promoted the fixation of aluminum oxide on the alloy's surface. Meanwhile, spraying aluminum oxide had almost no effect on corrosion resistance, which isn't surprising since the alloy itself contains a significant amount of aluminum.

Itrium plays an important role in forming a protective oxide film on the surface of the alloy because it increases the lattice constant and the diffusion of aluminum to the surface [21]. When studying the behavior of the alloy in a mixture of molten salts KCl-$MgCl_2$ at 700 °C with long-term exposure, it was found that volatile corrosion products likely form due to chlorination, as well as porous and loose products on the surface of the alloy that do not protect against corrosion [22].

In study [23], they investigated the corrosion of the AH14 alloy with a high aluminum content in a molten salt mixture of 45% NaCl and 55% $MgCl_2$ by weight at 600 °C. This alloy is planned to be used in the construction of salt-based nuclear reactors. Its composition is: Cr 22…27%, Al 3.5…4.5%, Fe 18…30%, C 0.4…0.5%, Ni balance. This alloy, in terms of aluminum content, is similar to Haynes 214, but the studies showed increased corrosion due to the destruction of the chromium carbide network,

magnesium penetration into the free space, and formation of the spinel $MgAl_2O_4$. Therefore, it is likely that this alloy is less resistant than Haynes 214.

Preparation for work in the SCW with steel samples. From 2012 to 2021, using supercritical water loops (SCWL) created at the NSC KIPT, three sessions of corrosion studies of reactor materials under irradiation were carried out. The duration of the sessions was 500…600 h [24, 25]. Samples of the materials under study were placed in four-channel irradiation chambers (IC) in special cassettes within a convective flow environment (24 MPa, 50 g/s) of subcritical and supercritical (up to 405 °C) water coolant under irradiation with 9 MeV electrons. To study the corrosion of the above-mentioned steels and alloys in the SCW environment, a convection loop "Loop 1d" of a similar design was made, the diagram and appearance of which are shown in Fig. 1.



a *b*

Fig. 1. Diagram (a) and photo (b) of the convection loop

Samples of Alloy 800, AL-6XN steels, and Haynes 214 alloy with dimensions 50.(1-4)x9.6x1.(1-9) mm are placed in IC. At the same time, a sample of zirconium ceramic can be tested [26]. A photo of the samples is shown in Fig. 2.

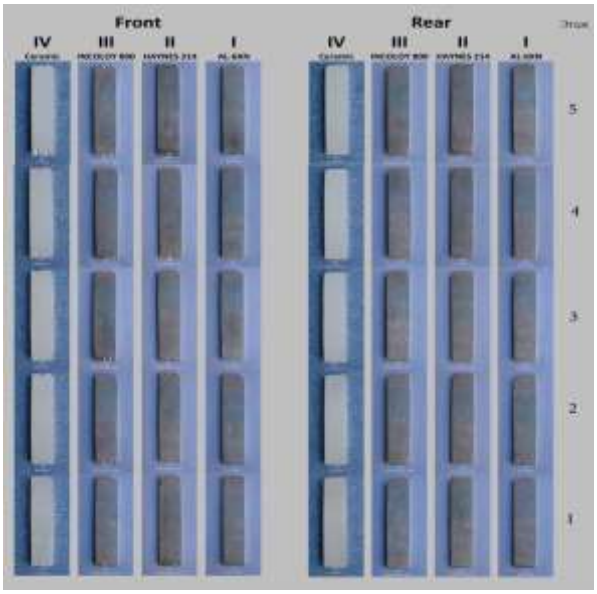


Fig. 2. Photo of the samples from the side of the electron beam impact (front) and from the opposite side (rear)

CONCLUSIONS

Alloy 800 and AL-6XN can be used for pretty much any purpose up to fairly high temperatures, while Haynes 214 alloy is really needed for operating products up to 1100°. It's worth doing corrosion tests to confirm the data from literature and to get new information for SCW conditions.

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СТАЛІ ДЛЯ РОБОТИ В НАДКРИТИЧНІЙ ВОДІ

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У надкритичній воді (НКВ) є сліди хлоридів, кисень, водень, які утворюються при термодеструкції води. Не можна виключати присутності перекісних сполук, які формуються навіть без дії радіації, судячи по окислювальній спроможності НКВ і без додавання окислювальних агентів. Отже, набір чинників, утворюючих корозійне середовище у НКВ, яка може використатися для охолодження реакторів, досить великій, і вимоги до матеріалів, які працюють у НКВ, високі.