

## STRUCTURE AND PROPERTIES OF AFA STEEL Fe-Ni-Cr-Al WITH VARIABLE ALUMINUM CONTENT

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Aluminum-containing austenitic steels (AFA steels), which may be promising for use as structural materials in nuclear reactors with liquid metal coolant, are investigated. Six ingots of alloys with variable aluminum content were obtained by the method of arc melting in an inert environment. After homogenizing annealing and cold deformation by rolling, samples for mechanical tests and structural investigation were made from the obtained strips, which were subjected to final annealing at the temperature standard for austenitic steels of 1050 °C. It was found that the maximum values of the yield strength and the ultimate tensile strength are observed at the aluminum content of 3.5 wt.%. It is significant that when the aluminum content is increased to 4 wt.%, the strengths and plasticity decrease. This behavior is associated with structural changes in steel.

### INTRODUCTION

Currently, the main structural reactor materials are steels of the austenitic and martensitic (ferritic-martensitic) classes. Ferritic-martensitic steels (EK 181, T91) with bcc lattice have sufficiently high radiation stability, but low mechanical characteristics at elevated temperatures, and also embrittle when interacting with liquid lead coolant. On the other hand, traditional reactor austenitic stainless steels (X18H10T, AISI 316L, and others) with fcc lattice have high mechanical properties and high corrosion resistance at elevated air temperatures. The main drawback of these steels is high swelling during irradiation, that is, low radiation resistance. The second major drawback of these steels is the high rate of dissolution in liquid lead or molten salts at temperatures higher than 500 °C.

Oxidation resistance is one of the main criteria that determine the durability of heat-resistant structural alloys. High resistance to oxidation is provided by the created external continuous oxide layer, which is thermodynamically stable, and further oxidation is limited by low counter-diffusion of metal or oxygen through this layer. For high temperature applications ( $\geq 600$  °C), the main oxides used to protect metal alloys are  $\text{Cr}_2\text{O}_3$  and  $\text{Al}_2\text{O}_3$ . But in many high-temperature environments, the  $\text{Al}_2\text{O}_3$  oxide layer provides a better degree of protection than  $\text{Cr}_2\text{O}_3$  [1–7]. The growth rate of the aluminum oxide layer is significantly lower than that of the  $\text{Cr}_2\text{O}_3$  layer, with the former being more thermodynamically stable. In addition, in an environment with water vapor, the key advantage of  $\text{Al}_2\text{O}_3$  over  $\text{Cr}_2\text{O}_3$  is greater stability [4, 5], which is relevant especially for thin-walled components such as heat exchangers. But when operating in an oxidizing environment at temperatures above  $\sim 600^\circ\text{C}$ , the presence of chromium and, as a result, the oxide layer  $\text{Cr}_2\text{O}_3$  gives an advantage and provides higher creep resistance [1–5].

Fe-Cr-Al based ferritic alloys capable of forming  $\text{Al}_2\text{O}_3$  oxide layers are also highly resistant to oxidation, but they are not suitable for structural applications

above  $\sim 600$  °C due to the low creep resistance resulting from their bcc crystalline structure.

This drawback is absent in aluminum-containing structural steels of the austenitic type with fcc lattice, in which a protective layer of aluminum oxide  $\text{Al}_2\text{O}_3$  is formed. These are the so-called AFA (Alumina Forming Austenitic) steels.

Currently, the composition of AFA steels is not optimized, and technological processes for controlling their microstructure and properties have not been developed. Therefore, the development of corrosion-resistant aluminum-containing austenitic reactor steels with a controlled microstructure and high radiation and mechanical characteristics is now a priority task for material scientists. Part of this task, namely: the study of the influence of the aluminum content in the composition of AFA Fe-Ni-Cr-Al on the structure and mechanical properties of this steel, was the subject of this work.

### 1. SELECTION OF COMPONENTS AND DEVELOPMENT OF THE PROCESS OF OBTAINING AFA STEELS

When developing AFA steels, it should be remembered that aluminum is a rather strong ferrite stabilizer, and the addition of chromium also stabilizes ferrite and at the same time reduces the aluminum content in the alloy, which is necessary for the formation of the  $\text{Al}_2\text{O}_3$  protective layer [8, 9].

Standard doping levels of  $\sim 4\text{--}6$  wt.% aluminum and  $\sim 10\text{--}25$  wt.% chromium in the alloys studied earlier can lead to a loss of stability of the austenitic structure of the matrix with a transition to a duplex ferritic-austenitic microstructure and a loss of creep resistance [6, 9, 10]. Stabilization of the austenitic matrix is achieved with the help of large additions of nickel, which leads to an increase in the price of the obtained alloys. Therefore, at the beginning of the research, the goal was to determine the optimal aluminum content at fixed concentrations of chromium (14 mass.%) and nickel (25 mass.%). In fact, attention was focused on studying the influence of aluminum content on the structure and mechanical properties of

the investigated alloys. Therefore, six alloys of the 61-xFe-25Ni-14Cr-xAl system were chosen for research, where the aluminum content was, respectively, x=2.5; 2.8; 3.0; 3.2; 3.5; 4.0 mass.%.

The specified steels were obtained by the method of arc melting in an environment of pure argon. The purity of the initial components was not lower than 99.9%. Studies of the microstructure of ingots have established that in order to achieve its homogeneity, at least five remeltings are required, with the ingots being turned over after each remelting. At the same time, in order to avoid evaporation of chromium, the melting had to be carried out at argon pressure in the chamber of at least 0.3 atm. The size of the obtained ingots was 6×15×60 mm.

Six ingots of aluminum-containing austenitic steels of different composition AFA-1 – AFA-6 were melted, namely (the content of elements, indicated below, is in mass percentage):

AFA-1 (Fe-25Ni-14Cr-2.5Al); AFA-2 (Fe-25Ni-14Cr-2.8Al); AFA-3 (Fe-25Ni-14Cr-3.0Al); AFA-4 (Fe-25Ni-14Cr-3.2Al); AFA-5 (Fe-25Ni-14Cr-3.5Al); AFA-6 (Fe-25Ni-14Cr-4.0Al).

For all obtained alloys, homogenizing annealing was carried out at temperature of 1200 °C for 3 h. As a result of such annealing, the alloys acquired a homogeneous microstructure with an average grain size of about 100 μm. For further research, the samples were rolled at room temperature with intermediate annealings at 1050 °C for 1 h. The total deformation by rolling was about 80%. At the final stage, tapes with a thickness of 1 mm were obtained, from which samples were prepared for further research.

## 2. RESULTS

### 2.1. X-RAY STRUCTURAL ANALYSIS OF SAMPLES

The XRD study of the samples was carried out on the X-ray diffractometer DRON-UM1 in copper Cu-K $\alpha$  radiation using a Ni selectively absorbing filter. Diffracted radiation was recorded by a scintillation detector.

For the study, samples AFA-1 – AFA-6 were taken after rolling to 1 mm and annealing at 1050 °C for 3 h.

Table 1

The phase composition of the samples

Sample	Phase	Lattice parameter, Å
1	fcc	3,600
2	fcc	3,597
3	fcc	3,596
4	fcc	3,596
5	fcc	3,594
6	fcc	3,593

All XRD patterns were subjected to standard processing (background separation, K $\alpha$ 1 doublet extraction, approximation of diffraction peaks by the pseudo-Voigt function) to obtain peak characteristics (diffraction angle  $2\theta$ , integral intensity  $I$ , integral width  $B$ , interplanar distance  $d$ ), necessary for further

calculations. The error in determining the lattice parameter of the fcc phase is  $\Delta a = 1 \cdot 10^{-3} \text{Å}$ .

The results of determining the phase composition of the samples are given in Table 1, the XRD patterns are presented in Fig. 1.

All test samples are single-phase and have an fcc structure. The diffraction peaks are very narrow, which indicates the coarse-crystalline state of the samples. The lattice parameter of the fcc phase increases with increasing aluminum content in the experimental alloys (Fig. 2). The intensity distribution of the diffraction lines of the fcc phase indicates the presence of texture in all samples. The predominant grain orientation varies from (200) (in samples AFA-3, AFA-6) to (220) (in sample AFA-4) and their combination (in samples AFA-1, AFA-2, AFA-5). Therefore, a clear dependence or correlation with the aluminum content is not observed (although here, perhaps, the large size of the grains and, as a result, their limited number in the analyzed area also contribute).

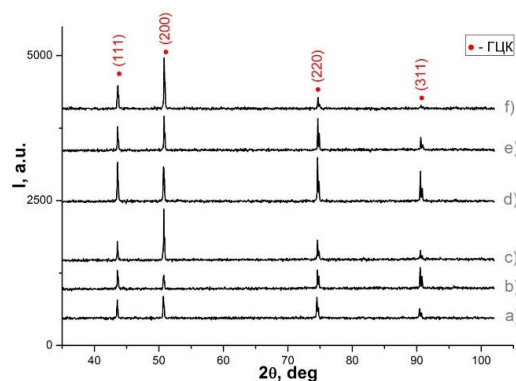


Fig. 1. XRD patterns of the samples: a – AFA-6 (Fe-25Ni-14Cr-4Al); b – AFA-5 (Fe-25Ni-14Cr-3.5Al); c – AFA-4 (Fe-25Ni-14Cr-3.2Al); d – AFA-3 (Fe-25Ni-14Cr-3Al); e – AFA-2 (Fe-25Ni-14Cr-2.8Al); f – AFA-1 (Fe-25Ni-14Cr-2.5Al)

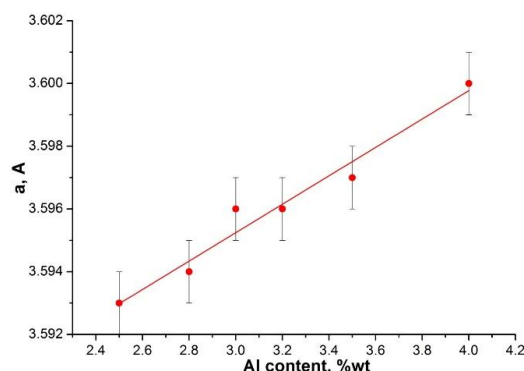


Fig. 2. Dependence of the lattice parameter of the experimental alloys on the aluminum content

### 2.2. STUDY OF MECHANICAL PROPERTIES OF STEELS

In order to evaluate the effect of aluminum content on the mechanical properties of the obtained steels, samples were prepared and their mechanical properties were studied during tensile loading at room temperature. The samples were made from the above-mentioned strips of alloys with a thickness of 1 mm. For each

composition of steel, two samples in the form of blades cut out by the electroerosion method, which were ground to the final dimensions, and then annealed at standard for austenitic steels temperature of 1050 °C for 3 h.

The dimensions of the test samples are given in Table 2. The length of the working part for all samples was 10 mm.

Table 2

Geometric dimensions of the samples

Steel	AFA-1	AFA-2	AFA-3	AFA-4	AFA-5	AFA-6
Al content, wt. %	2.5	2.8	3.0	3.2	3.5	4.0
Width, mm	1.93	2.00	1.93	2.03	1.98	1.87
Thickness, mm	0.94	0.96	0.96	0.96	0.96	0.94
Cross section, mm <sup>2</sup>	1.8142	1.92	1.8528	1.9488	1.9008	1.7578

Fig. 3 shows the tensile curves for all studied alloys, and Table 3 shows the results of processing these curves.

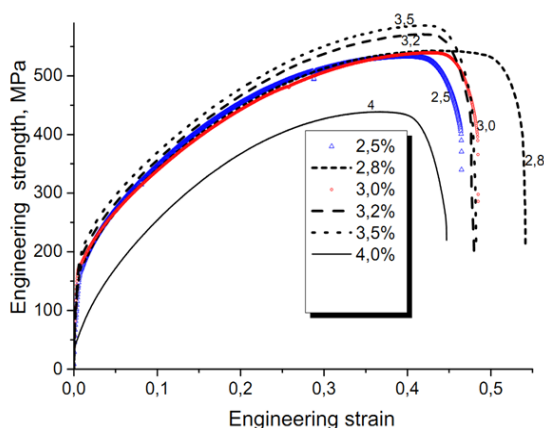


Fig. 3. Tensile loading curves of AFA steels with different aluminum content

These data indicate that yield strength  $\sigma_{0.2}$  and ultimate tensile strength  $\sigma_B$  of the samples increase by 26 and 10%, respectively, with an increase in the aluminum content  $x$  from 2.5 to 3.5 wt.% in the samples, while their plasticity (elongation to failure  $\delta$ ) changes in a non-monotonic way in the range 46...54%.

But it can be clearly stated that the content of 4 wt.% aluminum leads to a noticeable degradation of strength and plasticity. This behavior can be caused by structural changes related to the size, density, and coherence of B2 (NiAl) phase precipitates in samples with a high aluminum content [10].

Table 3

Mechanical properties of AFA steels with different aluminum content

Steel	AFA-1	AFA-2	AFA-3	AFA-4	AFA-5	AFA-6
Al content, wt. %	2.5	2.8	3.0	3.2	3.5	4.0
$\sigma_{0.2}$ , MPa	163.7	169	171.4	181.4	203.6	40
$\sigma_B$ , MPa	532.6	542.6	539	570.6	585.7	438
$\delta$	0.465	0.54	0.48	0.476	0.48	0.44

## CONCLUSIONS

1. In order to find out the effect of aluminum content on the structure and mechanical properties of aluminum-containing structural steels of the austenitic type with fcc lattice (so-called AFA steels), a study of the properties of Fe-25Ni-14Cr-xAl steel, where  $x=2.5; 2.8; 3.0; 3.2; 3.5; 4.0$  mass.%, has been performed. The technology of manufacturing steel ingots, homogeneous in composition and microstructure, has been developed.

2. X-ray structural analysis of steel samples revealed that they are single-phase and have an fcc structure. The lattice parameter of the fcc phase increases with increasing aluminum content.

3. Studies of the mechanical properties of the samples under tensile loading at room temperature indicate an increase in their strength characteristics, yield strength  $\sigma_{0.2}$  and ultimate tensile strength  $\sigma_B$ , by 26 and 10%, respectively, with an increase in the aluminum content  $x$  from 2.5 to 3.5 wt.%. This is accompanied by a non-monotonic change in their elongation to failure  $\delta$ , which has a rather large value, 46...54%. The introduction of 4.0 wt.% aluminum significantly reduces  $\sigma_{0.2}$  and  $\sigma_B$ , especially the yield strength: it is almost a quarter of the initial value (for  $x=2.5$  wt.%);  $\sigma_B$  being 82% of the initial value. At the same time, the plasticity  $\delta$  decreases by 5%. This may be caused by structural changes: the size, density, and coherence of B2 phase (NiAl) precipitates in samples with a high aluminum content.

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## **СТРУКТУРА ТА ВЛАСТИВОСТІ АФА СТАЛІ Fe-Ni-Cr-Al ЗІ ЗМІННИМ ВМІСТОМ АЛЮМІНІЮ**

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Досліджено алюмінійвмісні аустенітні сталі, які можуть бути перспективними для використання в якості конструкційних матеріалів у ядерних реакторах з рідкометалевим теплоносієм. Методом дугової плавки в інертному середовищі було отримано шість злитків сплавів із різним вмістом алюмінію. Після гомогенізуючого відпалу злитків та їх холодної деформації прокаткою із отриманих стрічок виготовлялись зразки для механічних випробувань та структурних досліджень, які зазнавали кінцевого відпалу за стандартної для аустенітних сталей температури 1050 °С. Встановлено, що максимальні значення межі плинності та межі міцності спостерігаються при вмісті алюмінію 3,5 мас.%. Показано, що при підвищенні вмісту алюмінію до 4 мас.% відбувається зменшення міцності і пластичності. Така поведінка пов'язується зі структурними змінами у сталі.