Development of photometric analysis of structural images in relation to the study of nickel-chromium alloy

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Abstract. This work presents preliminary results of the study of a new nickel-chromium corrosion-resistant alloy "Solution-N" intended to replace the imported G-35 alloy, which is currently used for the manufacture of chemical reactor vessels. In view of the limited amount of smelted material, photometric analysis of structural images is widely used to study its structure and properties. Based on the results of the studies carried out, it is proposed to choose the optimal structural state of the alloy, which has the highest corrosion resistance in the temperature range 600 - 650°C. Corrosion tests are currently being carried out in the laboratory of the prospective customer.

1. Introduction
Alloy "Solution-N" ("N") is intended for import substitution of nickel-chromium heat-resistant corrosion-resistant alloy G-35. At present, this alloy is used for the manufacture of vessels for chemical reactors, in which melts of transition metal chlorides were obtained in the temperature range 600 - 650°C. The factors that determine the operability of the reactor vessel material are intergranular corrosion, complicated by the effect of elevated temperature, and mechanical stresses from the column of transition metal chlorides. In 2019, in the pilot production of All-Union Institute of Aviation Materials (VIAM), by order of IMET RAS, an ingot of the experimental alloy "N" was smelted, the chemical composition of which was developed and patented by the applicant for IMET RAS, Mikhailov DL. Of the parameters characterizing the performance of the material under the operating conditions of the reactor, the intergranular corrosion speed is the most important.

2. Materials and Methods
Three sheets 6 mm thick and 21 × 40 mm in size were obtained from the melted ingot weighing 7.5 kg according to the developed three technology options. The purpose of the research stage of the "N" alloy in 2000 was to obtain preliminary data on the assessment of the structure and properties of the alloy on samples made from its sheets, manufactured according to the following options: rolling after hot pressing and open-die forging (I), repeated rolling after treatment (II), rolling from an ingot (III). For comparison, along with the investigated alloys, part of the work was carried out on alloys G-35 and Kh28Mo9N. The chemical composition of the alloys is shown in table 1.

| Table 1. Chemical composition of alloys (wt. %) |
|-----------------------------------------------|
| Alloy  | Cr       | Fe       | Ni       | Mo       |
|-------|----------|----------|----------|----------|
| G-35  | 32.90-32.95 | 1.14-1.22 | 57.31    | 7.98-8.06 |
| “N”   | 27.13-28.18 | 0.95-1.11 | 61.69-62.01 | 9.17-9.27 |
| Kh28Mo9N | 27.65-27.75 | 0.50-0.56 | 62.42-62.68 | 9.27-9.30 |
As can be seen from the table 1, the alloys are close in chemical composition, the differences in the concentrations of alloying elements do not exceed 0.5-4.75%. In addition to the elements indicated in the table 1, the alloys contain metallic and metalloid impurities in concentrations from 0.002-0.20%. It should be noted that boron in an amount of 0.002% in the G-35 alloy and lanthanum in an amount of less than 0.002% in the "N" alloy were introduced as alloying additives that affect the structure and diffusion processes in the alloys. The boron addition in alloy G-35 was not originally specified in the alloy certificate, but was detected in the alloy by photometric analysis of structural images. Comparison of the chemical compositions of the alloys shows that in the "N" alloy, the nickel content is adjusted upward to increase ductility, the chromium content is reduced, and the molybdenum content is reduced to give the alloy greater strength practically without a decrease in ductility.

Structural studies were carried out by light and electron microscopy, as well as by photometric analysis of structural images (PHASI) [1, 2], developed at IMET RAS. The main attention was paid to the "N" alloy, but the G-35 alloy as a reference for comparative analysis was studied by the same methods and on the same equipment as its domestic analogue. In (VIAM) [3], an ingot of the alloy "N", weighing 7.5 kg, was melted according to the proven technology. Three sheets with a thickness of 6 mm and dimensions of 21 × 40 mm were obtained from the ingot according to various technological options.

3. Melting and processing technology of the alloy "N"
Smelting of the alloy was carried out in an induction furnace in an argon atmosphere to prevent the evaporation of chromium at $T = 850 - 900^\circ C$ for 20 - 30 minutes heating up to $T = 1540 \pm 10^\circ C$ holding for 10 - 12 minutes, lowering the temperature to $1490 \pm 10^\circ C$ introduction of nickel-lanthanum alloy into the melt, stirring, pumping out argon and cooling with a furnace for 6 - 8 hours, provides an ingot with a chemical the composition given in table No 1. Smelting was preceded by preliminary heating of a clean charge in an induction furnace. From the melted ingot weighing 7 ± 0.25 kg, 3 sheets with the dimensions mentioned below were obtained in three different technological modes: 1) (I), obtained by hot pressing of a cast ingot ((I), preheated in a furnace to $T = 1140 \pm 10^\circ C$.

Pressing was carried out in a press in dies heated to $T = 950^\circ C$ in an induction device. Then there was carried out upsetting of the ingot into the butt end by 30-40 %, followed by all-round forging of the ingot and it was rolled on a Schmitz mill with a degree of deformation per pass from 10 to 15% and from 30 to 50% for heating. Rolling ended at a temperature not lower than 950°C. The sheet was obtained as a result of performing three rolling cycles, which alternated with heating up to $T = 1150^\circ C$.

On the first cycle, the sheet thickness was decreased from 40 mm to 25 ± 2 mm; on the second - after turning by 90°C, rolling to a thickness of 15 ± 2 mm; on the third, longitudinal rolling of sheets to a thickness of 6.0 ± 1.0 mm, also after turning by 90°C. Structural studies have shown texture in the rolling direction. The microstructure is mostly fine-grained (grain size is less than 10 microns, but there are individual grains up to 20 microns). Linear precipitations were found, most likely, of the secondary phase in the direction of rolling. 2) According to the option (II) was carried out up to a temperature of $1140 \pm 10^\circ C$. Hot pressing was carried out in the same mode as in option 1. It was followed by upsetting and drawing of an ingot with a degree of deformation from 30 to 40%, and machining of the slab. The ingot was cut into a sheet in three steps. Rolling was carried out on a Schmitz rolling mill with a degree of deformation per pass from 10 to 15% and from 30 to 50% for heating. The rolling temperature was not lower than 950°C. At the first stage of rolling, the slab was brought from a thickness of 40 mm to a thickness of 25 ± 2 mm. Between the stages of rolling, heating was carried out to a temperature of $1140 \pm 10^\circ C$. At the second and third stages of rolling, the sheet was brought successively to a thickness of 15 ± 2 mm and finally to a thickness of 6.0 ± 1.0 mm. The study of the microstructure in the mode without all-round forging showed the presence of texture in the direction of rolling. The microstructure is predominantly fine-grained (grain size less than 10 microns), but there are individual grains up to 50 microns. There were also line segregations, most likely, of the secondary phase in the direction of rolling. 3) According to this version (I), hot rolling was carried out in the cast state on the Schmitz mill with a deformation rate of up to 10% per pass and 40% per heating. The temperature of the end of rolling is not lower than 950°C. At the first stage, a
sheet with a thickness of 25 ± 2 mm was obtained from a rolled billet 40 mm thick. At the next stage, the sheet with a thickness of 25 ± 2 mm was rolled by rolling to a thickness of 15 ± 2 mm, and at the final stage the sheet was brought to a thickness of 6.0 ± 1.0 mm. Between each stage of rolling, the billets were heated up to $T = 1140 ± 10^\circ C$ and tilting at $90^\circ C$. Investigations of the microstructure in the mode without all-round forging showed the presence of texture in the direction of rolling. The microstructure is predominantly fine-grained (grain size less than 10 $\mu\text{m}$), but individual grains up to 50 $\mu\text{m}$ are present. There are also line segregations, apparently of the secondary phase in the direction of rolling. After completing rolling, the sheet was mechanically descaled and polished. Figure 1 shows a comparison carried out using PHASI of typical microstructures of the developed alloy, formed by processing according to the considered technological options.

![Figure 1](image_url)

**Figure 1.** The results of the PHASI analysis of the "N" alloy after treatments (III) and (I): on the left is the image of a fragment of the sample and the brightness spectrum of the reflection of visible light from its surface in the (III) state, on the right - the same, but for the (I) state.

PHASI is a software-analytical complex in which the structure of a reference fragment is compared according to a differential scheme (in our case, the structure obtained by variant (III) was chosen as the standard) with fragments of structures obtained by processing (I) and (II). Compared as images of fragments and the brightness spectra of the reflection of visible light from them. The latter are presented in the coordinates: "intensity of light reflection $I$ - spectral reflection density $p(I)$". The reflection intensity is measured on a linear scale, the zero value of which is taken to be the state of the surface of the fragment with complete absorption of the incident light flux, and as unity, the state of its total reflection. The ratio of the number of pixels having a reflection intensity in a given interval $I$ to the total number of pixels into which the fragment image is divided is taken as a measure of the spectral density. On the abscissa axis, you can select intervals using color coloring and transfer the colors of the selected intervals to the images of fragments, thereby visualizing the structure elements that contribute to the reflection of light in the selected interval.

4. **Results and discussion.**

Figure 2 shows that in the (III) state the "N" alloy in the structural state is practically homogeneous, in the (I) state it consists of two structural components, and in the (II) state of three. In all states, the
structural component, colored green, prevails. To determine the phase composition of structural components, standards are needed, which we do not yet have.

![Figure 2](image)

**Figure 2.** The results of the PHASI analysis of the "N" alloy after the (III) and (II) treatments: on the left is an image of a sample fragment and the brightness spectrum of the reflection of visible light from its surface in the (III) state, on the right - the same, but for the (II) state.

Correlation analysis showed that the correlation coefficient between the hardness data of the studied nickel-chromium alloys and the grain sizes is $C = 0.943$, which indicates the possibility of analytically describing the dependence $H - H_c = f (D - D_c)$ with a high degree of reliability. Figure 3 shows this dependence plotted from experimental data (solid line) and from power-law approximation data (dashed line). The latter is analytically described by the equation: $(H - H_c = 0.439(D - D_c)^2 + 5.589 (D - D_c) - 343.31$, with a confidence factor $R^2 = 0.998$, where: $H$ is the Brinell hardness, $D$ is the average grain size, $H_c$, $D_c$ are the average values of these parameters. Figure 4 shows the results of a comparison by the PHASI method of the structures of the "N" alloy in the states formed by treatments according to the options I, II and III.

Table 2 shows the treatment modes alloy "N" used for the formation of various structural components. It can be seen from the presented data that the PHASI method allows you to clearly distinguish the structural states of the alloy after various treatments. The table in the last column shows the data on the energy accumulated in the sample in relative units, obtained by the PHASI method.

**Table 2.** Heat treatment modes and its results

| "N" alloy samples | Quenching and medium T, °C | Cooling mode | Quenching and medium T, °C | Cooling mode | $H_c$ | $U$ – energy in relative units |
|-------------------|----------------------------|--------------|----------------------------|--------------|------|-------------------------------|
| 3                 | 1107                       | 20 water     | water                      | 800          | 30 air | 187                           | 0.5667 |
| 6                 | 1121                       | 15 water     |                            |              |       | 187                           | 0.5054 |
| 5                 | 1125                       | 20 water     |                            |              |       | 184                           | 0.5394 |
| 4                 | 1107                       | 25 water     |                            |              |       | 180                           | 0.6430 |
| 2                 | 1107                       | 25 water     |                            |              |       | 187                           | 0.5834 |
| 1                 | 1107                       | 30 water     |                            |              |       | 184                           | 0.5247 |
Figure 3. Dependence (Нв -f(D -)) for nickel-chromium alloys, where:

Нв - hardness according to Brinell, D - grain diameter (μm)

(a)                                             (b)                                          (c)

Figure 4. Typical structures of the "N" alloy in the states formed by treatments according to the options: a – (III), b – (I), c – (II) × 110

Figure 5 shows the results of a comparative analysis of the alloy under study in states (1) and (3) table 2. In the upper part there are images of fragments, in the lower part of the figure - the brightness spectra of reflection of visible light, taken from them. Correlation analysis was carried out in order to establish the relationship between the size of the areas colored in the corresponding range of the spectrum and the values of the Brinell hardness and the values of the energies accumulated in the structural components of the alloy, colored in the colors of the spectrum. The analysis showed that there is no correlation between the areas and hardness values, but revealed it for the data arrays of energies and areas occupied by the structural component, colored in the colors of the alloy matrix and black. In the first case, a negative correlation was found (correlation coefficient C = 0.223). In the second, a positive correlation was found with a correlation coefficient C = 0.8082.

Figure 6 shows the structures of the G-35 alloy obtained by the PHASI method for the base metal and the weld under the same shooting conditions. Differences in the ground state obtained as a result of complex mechanical-thermal treatment and the cast structure of the weld metal are clearly visible. Welding and heat treatment promotes coagulation of boron with the formation of globular discharge.
5. Conclusions

1. A new domestic heat-resistant corrosion-resistant alloy "N" has been developed, which is not inferior in properties to the imported alloy G-35 (USA).

2. A preliminary study was carried out to select its optimal state according to the data of photometric analysis.

3. The presence of boron in the G-35 alloy was established and the nature of its distribution in the base metal and weld was studied.

4. The dependence of hardness on the average grain size in the investigated nickel-chromium alloys has been established.

5. PHASI method was successfully used for testing intergranular corrosion.
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