Досліджено технологічний процес одержання сплаву на основі заліза з ефектом пам'яті форми, обґрунтований вибір хімічного складу сплаву, обрані режими термічної обробки. Показано, що запропонований сплав має високий ступінь відновлення форми при збереженні таких важливих властивостей, як міцність, в'язкість, корозійна та окалиностійкість. Отримані математичні моделі впливу хімічного складу сплаву на межу міцності сплаву і значення ефекту пам'яті форми

Ключові слова: сплав на основі заліза, ефект пам'яті форми, механічні властивості, окалиностійкість, корозійна стійкість

Исследован технологический процесс получения сплава на основе железа с эффектом памяти формы, обоснован выбор химического состава сплава, выбраны режимы термической обработки. Показано, что предложенный сплав имеет высокую степень восстановления формы при сохранении таких важных свойств, как прочность, вязкость, коррозионная и окалиностойкость. Получены математические модели влияния химического состава сплава на предел прочности сплава и значения эффекта памяти формы

Ключевые слова: сплав на основе железа, эффект памяти формы, механические свойства, окалиностойкость, коррозионная стойкость

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DEVELOPMENT OF AN IRON-BASED ALLOY WITH A HIGH DEGREE OF SHAPE RECOVERY

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1. Introduction

Alloys with a shape memory effect (SME) are the alloys that "remember" geometry. These properties are the result of phase martensite-austenitic transformations [1]. Martensite phase transformation of alloys can be caused by other factors as well, but they mostly depend on temperature [2]. In the austenite phase, shape-memory alloys are a hard and solid material, and when these alloys are in the state of martensite – they are soft and flexible. In the martensite state, shape-memory alloys can stretch or deform under the influence of an external force [3–6]. As soon as the heating occurs, an alloy will be transformed into the state of austenite and recover any section, which was deformed [7–9]. The force acting on SME alloys after compression can be employed to perform such tasks as turning on or off devices, launching or closing a facility. SME alloys can be used for a welding-free joint of structures, fuel supply lines, high-pressure pipelines, controlling elements in the form of couplings, springs, etc.

However, these alloys are quite expensive and their application in mechanical engineering is not economically expedient [10–11]. Given this, studies into finding and developing new alloys with a shape memory effect are relevant now; in this case, of the largest interest for metallurgy and mechanical engineering are the iron-based alloys. When designing appropriate alloys, it is necessary to take into account a relation between high mechanical and performance properties and sufficient values of coefficients that describe the shape memory effect.

2. Literature review and problem statement

Over the past two decades, inexpensive alloys based on the system iron-manganese-silicon with a shape memory effect have attracted considerable attention. These alloys are considered to be a cost-effective alternative to expensive alloys based on nickel and titanium [12]. A shape memory effect relates to a phenomenon under which the alloys after deformation should return to their previous shape when heated. This group of alloys can be used for strengthening reinforced concrete beams. Their advantage is the ability to greatly reduce the possibility of damage due to corrosion, fire, vandalism, mechanical damage and aging. Article [13] proposes to replace the fibers of reinforced polymers with the strips of alloy based on iron with SME, which can be applied for strengthening reinforced concrete beams.

Studies into alloys based on Fe–Mn–Si [14] have shown that the degree of reversibility of the motion of dislocations could be increased by obtaining the energy of packing defect until about 20 mJ m^{-2}. This is achieved by the correlation of chemical composition of the alloy.

Shape memory alloys based on iron are the materials, which also have a large potential for use in civil engineering structures. This is especially true of alloys of the system iron-manganese-silicon; however, their application is still under development. Changes in the composition of an alloy and in the production parameters imply new prospects of application, especially in the field of repair of structures. Paper [15] presents the basics of martensite transformation from an engineering point of view, as well as some key properties, such as durability, recovery, corrosion resistance, weldability and workability.

Materials based on the iron-manganese alloys were also considered as orthopedic implants. Article [16] explored the alloy FeMn30, in particular its micro structural, mechanical and corrosion characteristics. The results obtained were compared with iron. It was shown that the alloy FeMn30 consists of phases of antiferromagnetic γ-austenite and ε-martensite and possesses better mechanical properties than iron and steel 316L.

Known alloy "Nitrogen-containing alloys based on iron that have the properties of damping and a shape memory effect" [17] is a high-alloy manganese austenitic steel with a possible dispersion hardening by carbon or nitrogen. The alloy has the following chemical composition (% by weight): 5–50 Mn; 0.01–0.8 N; possibly 0.1–20.0 Cr; 0.1–20.0 Ni; 0.1–20.0 Co; 0.1–8.0 Si; 0.1–3.0 Cu; 0.1–1.0 V; 0.1–1.0 Nb; 0.1–3.0 Mo; 0.001–1.0 C; 0.0005–0.02 rare earth metals; the rest is iron. A shortcoming of this steel is insufficient magnitude of SME and low strength characteristics. The low properties are due to the absence of an effective dispersion hardening, which is caused by the lack of strong carbide-forming (nitride-forming) elements, or the amount of these elements is very small (to 0.8 %).

The drawbacks of "Non-magnetic steel" [18], the dispersion-hardening austenitic steel 20G18S2F with a shape memory and carbide strenghtening, which contains the elements (% by weight): 0.15–0.25 C; 17.0–19.0 Mn; 2.0–2.5 Si; 1.1–1.5 V and the rest is Fe, are the insufficient value of SME and low strength. This is caused by the lack of carbide dispersion hardening due to the reduced amount of carbon at smaller quantity of carbide-forming elements. This state prevents increasing the magnitude of effort to unbend when implementing SME, which reduces the functionality of steel.

"Dispersion-hardening austenitic steel with a shape memory" [19] contains the elements (% by weight): carbon 0.25–0.5; manganese 16.0–25.0; silicon 1.0–3.0; vanadium 1.1–3.0; niobium 0.1–1.0; tungsten 0.1–3.0; the rest is iron. In this case, the ratio (V+Nb) to carbon should be not less than 6 %, and the total amount of carbide-forming elements (V+Nb+W) – not less than 1.8 %. In this case, steel can additionally contain 0.1–3.0 % by weight of molybdenum. The main and significant shortcomings of the steel are insufficient mechanical properties.

Thus, the above-considered alloys based on iron with SME have a number of unresolved issues, in particular, they do not always provide for a sufficient magnitude of SME coefficient while maintaining a high level of operational performance.

Therefore, it is a promising direction to create a new alloy based on iron with SME with a substantiated selection of chemical composition of the alloy to ensure high values of operational properties.

3. The aim and objectives of the study

The goal of present work is to develop a new iron-based alloy with a shape memory effect.

To accomplish the set goal, the following tasks have to be solved:

– to examine the technological process of obtaining an iron-based alloy with a shape memory effect;

– to explore the mechanical properties and the performance characteristics of the resulting alloy;

– to receive mathematical models of the impact of chemical composition of the alloy on tensile strength of the alloy and values of a shape memory effect.

4. Material of research, technological modes of receiving an iron-based alloy with a shape memory effect and techniques for conducting the research

4. 1. Examined material that was used in the experiment

The set task is solved based on the fact that an iron-based alloy with a shape memory effect contains iron, manganese, silicon, carbon, vanadium, niobium, tungsten, aluminum, copper, nickel, chrome, sulphur and phosphorus. The ratio of components (% by weight) is as follows: manganese 12.0–17.0; silicon 1.0–4.0; carbon 0.2–0.7; vanadium 1.0–4.0; niobium 0.05–1.0; tungsten 2.0–4.0; aluminum 0.1–0.2; copper 0.05–0.5; nickel 0.05–0.5; chrome 0.1–0.5; sulfur to 0.0025; phosphorus to 0.003; the rest is iron.

4. 2. Technological modes of receiving an iron-based alloy with a shape memory effect

The alloy smelting was carried out in a vacuum induction furnace of the type OKB-862 (Russia).

The samples were exposed to the following thermal treatment [20]: annealing at temperatures of 900 °C at varying duration depending on the dimensions of the samples. Then we conducted hardening at a temperature of 1150 °C (a single-stage heating at 800 °C for 10 minutes immediately before hardening, hardening at 1150 °C of varying duration depending on the dimensions of the samples, subsequent cooling in open air). The final strengthening thermal treatment was aging under two modes:

1) temperature 1000 °C for one hour followed by cooling in the open air;

2) temperature 800 °C for ten hours followed by cooling in the open air.

4. 3. Research methods and modeling the values of tensile strength of the alloy and a shape memory effect

Mechanical tests were conducted at room temperature in line with GOST 1497-84 on a universal machine that complies with GOST 28840-90. Standard samples for tensile test underwent testing at room temperature; length of the samples was 100 mm.

A visual study of the scale resistance involved heating the samples to 600–1000 °C with a 50 °C step in the open air and subsequent examination of the surface.

Experiment on corrosion resistance [21] of the alloy was conducted by weight method in a 10 % solution of sulphuric acid.

Quantitative phase analysis for residual austenite in the alloy was carried out using the x-ray machine DRON-3 (Russia) according to the standard procedure [22].

The simulation was carried out in the MathCAD programming package, plotting the graphs and nomograms was executed in the Excel 2010 software [23].

5. Experimental data and processing of the obtained results of experiment

5. 1. Results of experiments on the process of receiving an iron-based alloy with a shape memory effect

The proposed alloy differs from the known iron-based alloys with a shape memory effect by the additional introduction of aluminum, copper, nickel, chromium, sulphur and phosphorus. Alloying the steel can significantly improve performance efficiency with an increase in the shape recovery factor in the presence of a minimal amount of expensive alloying elements in the alloy.

One of the important moments when obtaining a dispersion-hardening iron-based alloy is the deoxidation. The most common steel deoxiders include silicon, manganese, and aluminum. Steel deoxidation is promoted by treating with vacuum (steel vacuum processing). During steel deoxidation with aluminum, a rather strong oxide of Al_2O_3 forms, which is released in the liquid metal in the form of a separate solid phase. Aluminum is typically employed with manganese and silicon to give the aluminum oxide a chance to bind to thin liquid slag.

Alloying with copper implies creating conditions for the formation of austenitic structure of alloys, as well as to improve their corrosion resistance.

The purpose of chrome alloying is to reduce the energy of a packing defect of austenite and to improve corrosion resistance of alloys. At chromium content less than 0.1 % by weight, the listed indicators cannot be achieved.

Nickel is a very strong austenite-forming element, which is added for the purpose of creating an austenitic maternal phase, as well as to improve the corrosion and scale resistance of alloys. The desired effect cannot be achieved at nickel content less than 0.05 % by weight.

To improve the mechanical properties and magnitudes of SME, it is necessary to stabilize austenite relative to the formation of martensite of cooling, in this case, it is required to preserve the ability of steel to form the martensite of deformation at 20 °C. This result is achieved by the additional alloying of the alloy with strong carbide-forming elements. Alloying the alloy with silicon reduces the energy of packing defect of austenite and increases its yield strength, which has a positive effect on the properties of a shape memory. In addition, silicon additives in the range of 1.0–4.0 % increase corrosion resistance and scale resistance of the material. Carbon is the austenite-forming element, which is added to alloys with the aim of creating the austenitic structure before deformation. In addition, carbon strengthens both austenite and martensite, which also exerts a positive effect on the properties of a shape memory. Manganese is added to alloy as the austenite-forming element, as well as the element that increases the solubility of impurities of introduction in the alloys, which in turn are austenite-forming, and thus it provides austenite state of the alloy before deformation.

The essence of chromium alloying is to reduce the energy of a packing defect of austenite and to improve corrosion resistance of the alloys. Nickel is a very strong austenite-forming element and it is added with the aim of creating an austenitic phase, as well as to improve corrosion and scale resistance of the alloys. The content of cobalt in the alloy also contributes to the formation of austenite phase and improves hot workability of the alloys. Alloying with copper is conducted to create conditions for the formation of the austenitic structure of alloys and to improve their corrosion resistance. Molybdenum decreases the energy of a packing defect of austenite and thereby contributes to the formation of martensite at deformation, as well as improves scale resistance of the alloy.

Vanadium and niobium are added to increase the yield strength of alloys and to improve the solubility of elements of introduction in the liquid phase of alloys. The simultaneous presence of vanadium and niobium in the alloys makes it possible to control the content of carbon in a solid solution using the thermal treatment methods. Increasing or decreasing the content of alloying elements beyond the recommended ranges significantly degrades performance efficiency of the goods made of the proposed alloy.

Aging the proposed alloy makes it possible to considerably increase the strength characteristics and, at the same time, to increase the magnitude of SME. In the process of release of carbides at aging, an austenitic matrix is partially freed from carbon and carbide-forming elements and becomes capable of forming even larger amount of martensite of deformation, which determines the magnitude of SME. After SME manifests itself when heated, the austenitic steel remains resistant to the formation of martensite of cooling and stores unchanged the obtained deformation in a wide range of temperatures (up to 500 °C).

Changes in the values of characteristics of alloy strength depending on the modes of thermal treatment are given in Table 1.

Table 1

Mechanical characteristics of alloy

Number of mode of alloy treatment	$\sigma_{0.2}$, MPa	σ_B, MPa	δ, %	ψ, %
1	700–930	1180–1310	17–25	21–27
2	720–1190	1200–1380	23–32	28–36

Results of examining scale resistance revealed that when the samples were heated in the temperature range of 600–1000 °C, surface oxidation was not observed.

In the course of conducting experiment on the corrosion resistance of alloy, it was found that the alloy was corrosion resistant and was not inclined to change the weight when exposed to a 10 % solution of sulfuric acid.

Study of the microstructure confirmed the existence of dispersion hardening in the alloy following the aging regimes; in this case, after the second treatment mode, there were more carbide impurities than after the first treatment mode.

Diffractogram of the alloy after hardening at a temperature of 1150 °C and cooled in the open air showed a surge corresponding to γ-Fe, therefore the content of residual austenite in the alloy is 100 %.

We used hot rolling to thin the samples. Rolling was held in three stages:

1. Heating to 500 °C and remaking in the forge to a thickness of about 30 mm.
2. Heating to 800 °C, rolling to a thickness of 2.7 mm.
3. Heating to 1190 °C, rolling to a thickness of 2.0–2.1 mm.

Properties of the shape recovery were measured by tensile tests of the samples with a thickness of 2.0–2.1 mm and a length of 30 mm. The samples were deformed by 5 % at room temperature, and, after that, heated above the temperature of the reverse martensite transformation. The degree of shape recovery (α) was estimated by formula:

$$\alpha=((l_d-l_h)/(l_h-l_0))\cdot 100\ \%, \qquad (1)$$

where l_0 is the initial length of the sample; l_d is the sample length after deformation; l_h is the sample length after heating.

Research results demonstrated that the degree of shape recovery of the proposed alloy is 78–97 %.

We also explored the proposed alloy after hardening and aging in line with mode 2. By using hot rolling, we obtained in advance the plates of thickness 1.35 mm, width of 10 mm, and length of 200 mm. The plates after thermal treatment were bent in a ring at room temperature. A manifestation of the shape memory effect was observed when the rings were placed in the furnace preheated to 500 °C. Table 2 gives the results obtained.

Received data (Table 2) showed that the proposed alloy with different composition exhibited quite high values (125–160°) of a shape memory effect, which was characterized by the angles of unbending the samples (φ).

The proposed alloy is plastic enough; it can undergo hot, warm and cold deformation in the open air.

Therefore, the proposed alloy possesses a high degree of the shape recovery while maintaining such important properties as strength, toughness, corrosion and scale resistance.

5. 2. Mathematical modeling of results of experiments after combined processing

Based on received data (Table 2), we constructed dependences (Figs 1–22) of a value of the shape memory effect and tensile strength on chemical composition of the alloy in the normalized form [24–28]. Experimentally obtained values of chemical composition of the alloy for each element are valid values (Table 2), which for the purpose of subsequent construction of mathematical models were converted to the normalized form. In this case, the maximal valid value of a chemical element was accepted as “+1”, the minimal as “–1”.

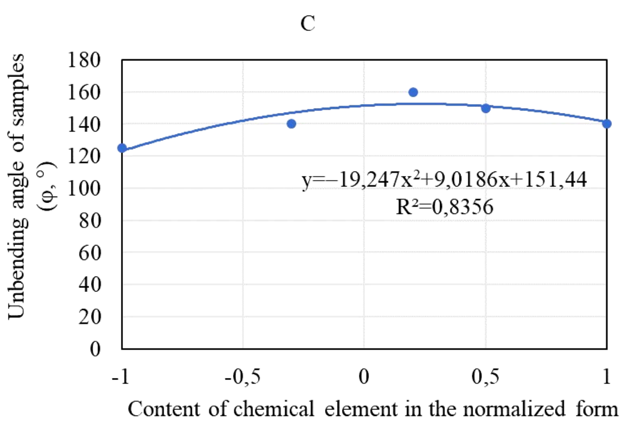

Fig. 1. Dependence of unbending angles of the samples (φ) on the content of carbon in the obtained alloy in the normalized form

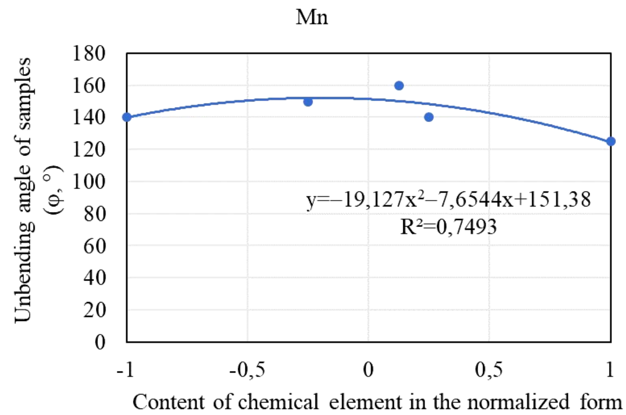

Fig. 2. Dependence of unbending angles of the samples (φ) on the content of manganese in the obtained alloy in the normalized form

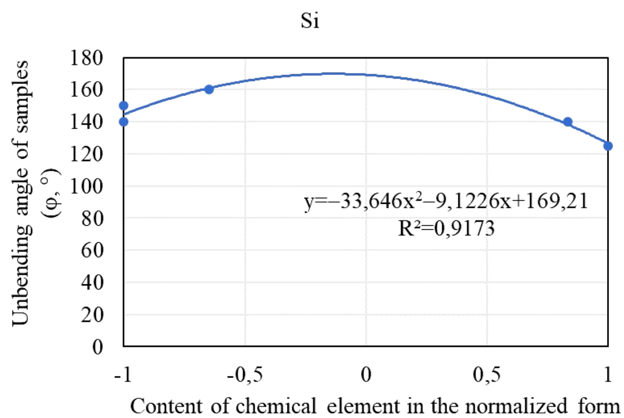

Fig. 3. Dependence of unbending angles of the samples (φ) on the content of silicon in the obtained alloy in the normalized form

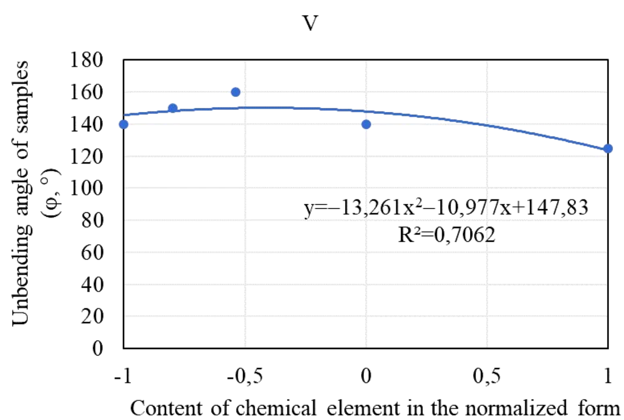

Fig. 4. Dependence of unbending angles of the samples (φ) on the content of vanadium in the obtained alloy in the normalized form

Table 2

Composition, values of a shape memory effect (φ), tensile strength of the proposed alloy

Steel	C	Mn	Si	V	Nb	W	Fe	Al	Cu	Ni	Cr	φ, °	σ_B, MPa
1	0.35	14.2	2.2	3	0.2	3.5	the rest	0.17	0.1	0.3	0.4	125	1200
2	0.42	13.9	2.1	2.5	0.1	3.32	the rest	0.15	0.08	0.1	0.35	140	1270
3	0.47	13.85	1.21	2.23	0.085	3.25	the rest	0.15	0.32	0.25	0.23	160	1380
4	0.5	13.7	1	2.1	0.07	3.1	the rest	0.16	0.2	0.2	0.2	150	1310
5	0.55	13.4	1	2	0.05	2.8	the rest	0.15	0.12	0.08	0.18	140	1280

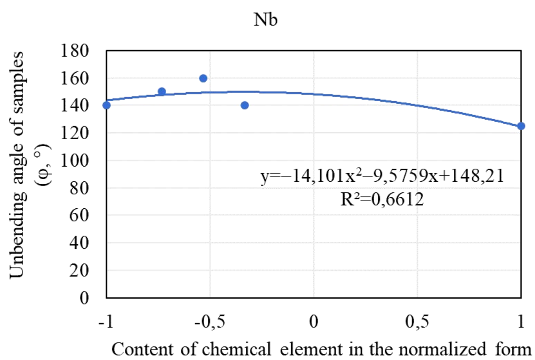

Fig. 5. Dependence of unbending angles of the samples (φ) on the content of niobium in the obtained alloy in the normalized form

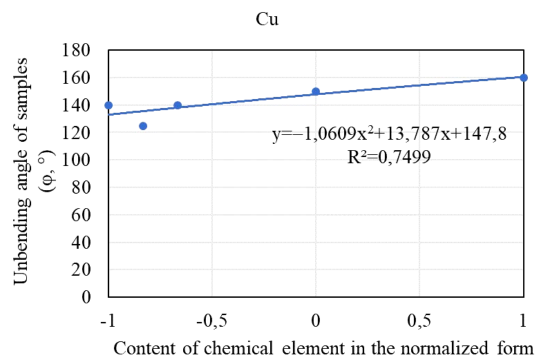

Fig. 9. Dependence of unbending angles of the samples (φ) on the content of copper in the obtained alloy in the normalized form

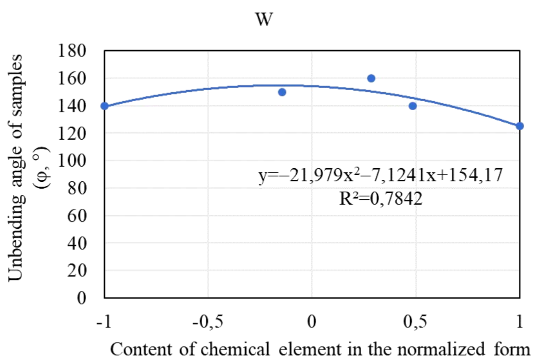

Fig. 6. Dependence of unbending angles of the samples (φ) on the content of tungsten in the obtained alloy in the normalized form

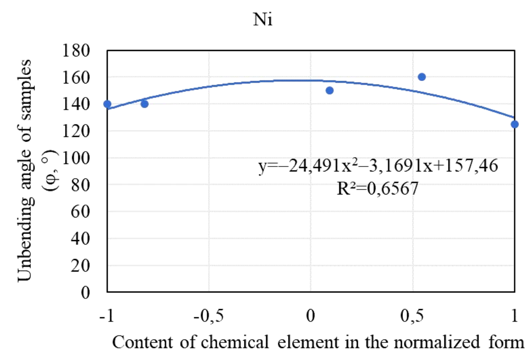

Fig. 10. Dependence of unbending angles of the samples (φ) on the content of nickel in the obtained alloy in the normalized form

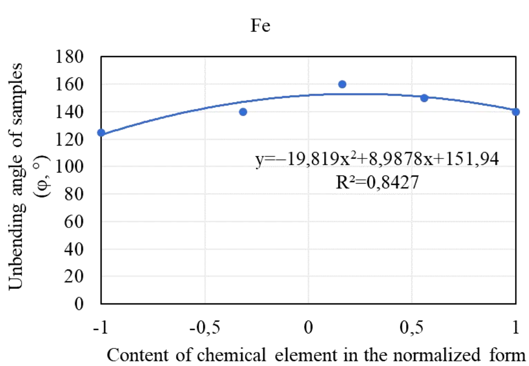

Fig. 7. Dependence of unbending angles of the samples (φ) on the content of iron in the obtained alloy in the normalized form

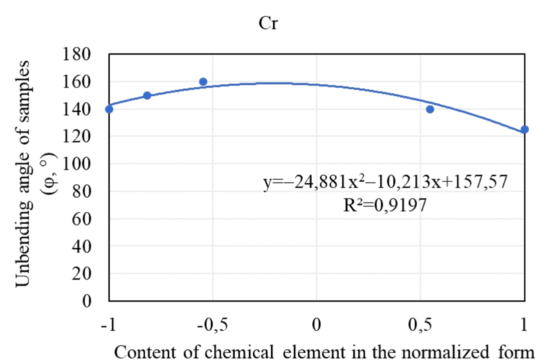

Fig. 11. Dependence of unbending angles of the samples (φ) on the content of chromium in the obtained alloy in the normalized form

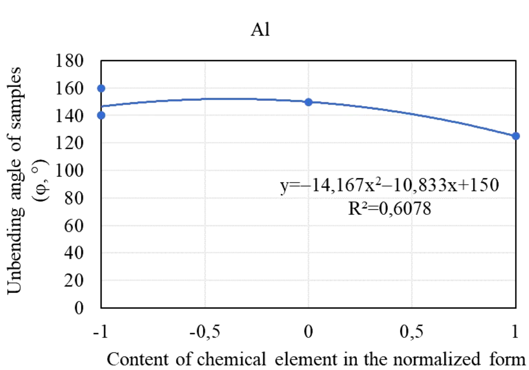

Fig. 8. Dependence of unbending angles of the samples (φ) on the content of aluminum in the obtained alloy in the normalized form

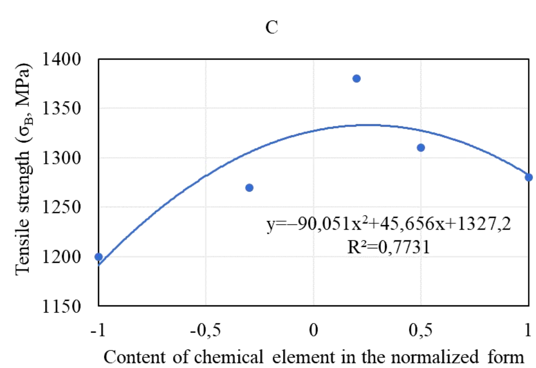

Fig. 12. Dependence of tensile strength on the content of carbon in the obtained alloy in the normalized form

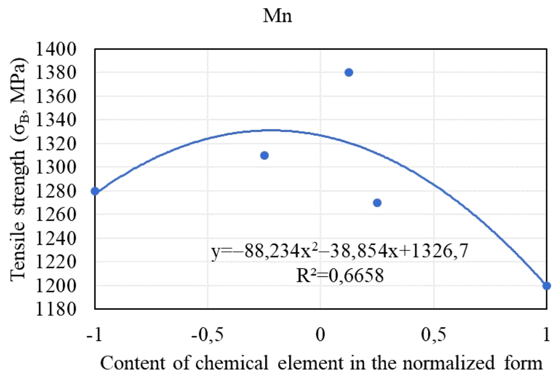

Fig. 13. Dependence of tensile strength on the content of manganese in the obtained alloy in the normalized form

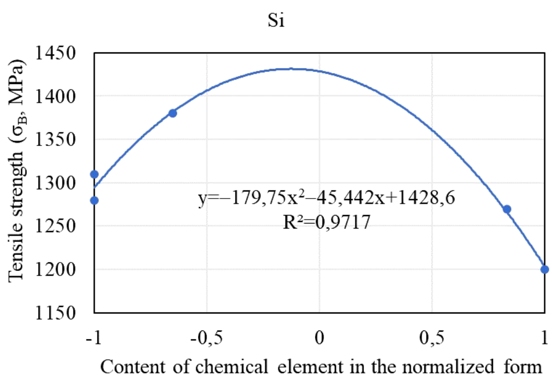

Fig. 14. Dependence of tensile strength on the content of silicon in the obtained alloy in the normalized form

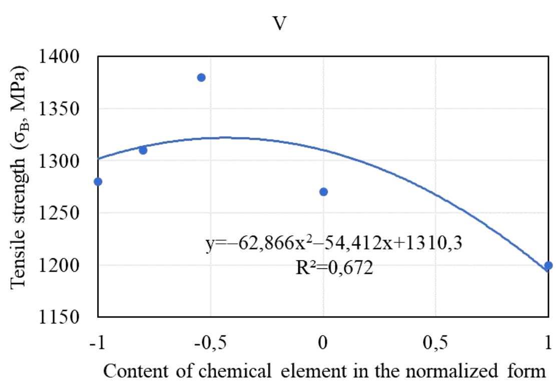

Fig. 15. Dependence of tensile strength on the content of vanadium in the obtained alloy in the normalized form

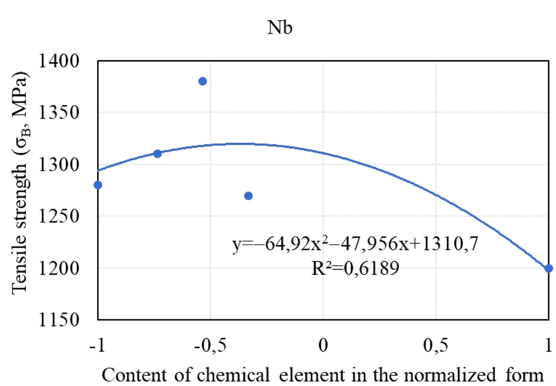

Fig. 16. Dependence of tensile strength on the content of niobium in the obtained alloy in the normalized form

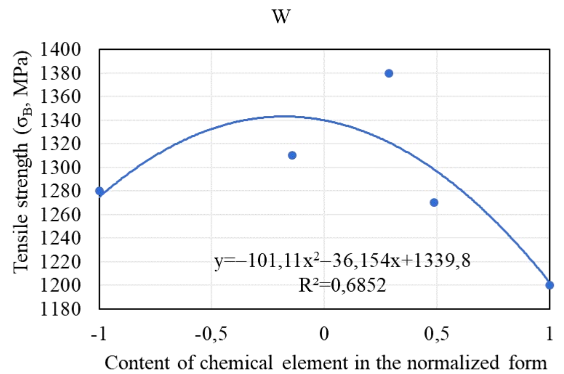

Fig. 17. Dependence of tensile strength on the content of tungsten in the obtained alloy in the normalized form

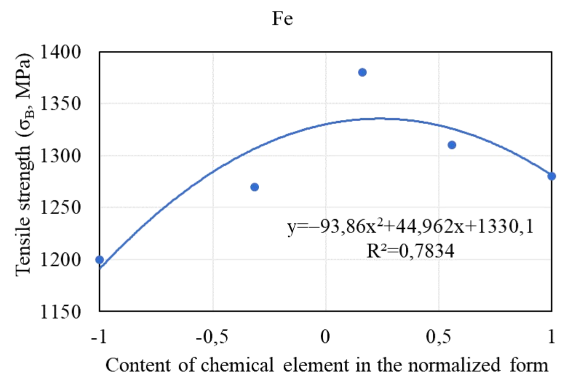

Fig. 18. Dependence of tensile strength on the content of iron in the obtained alloy in the normalized form

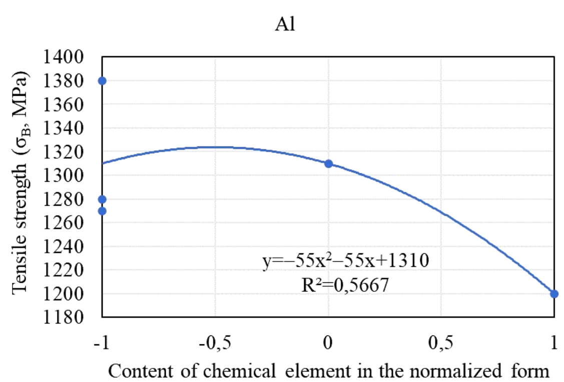

Fig. 19. Dependence of tensile strength on the content of aluminum in the obtained alloy in the normalized form

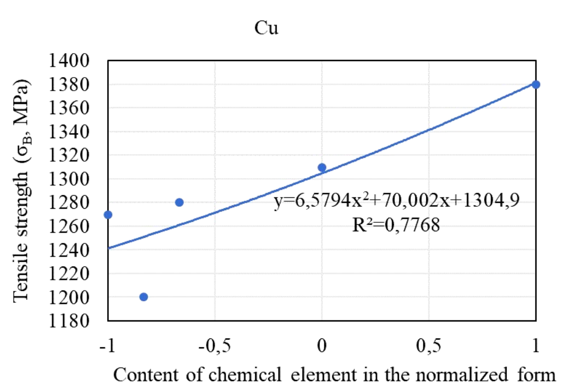

Fig. 20. Dependence of tensile strength on the content of copper in the obtained alloy in the normalized form

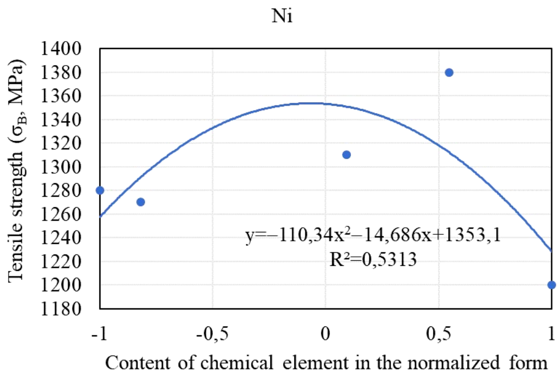

Fig. 21. Dependence of tensile strength on the content of nickel in the obtained alloy in the normalized form

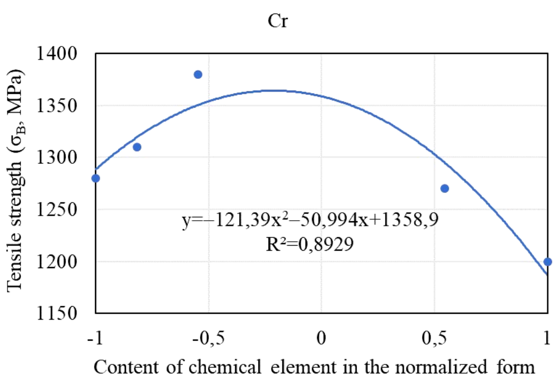

Fig. 22. Dependence of tensile strength on the content of chromium in the obtained alloy in the normalized form

Received dependences (Fig. 1–22) allowed us to obtain mathematical models of the effect of each chemical element on the properties of the alloy. The models were received by using the second-degree polynomial approximation method with the verification of adequacy of the model by the magnitude of approximation credibility (R^2).

6. Discussion of modeling results

We received a new iron-based alloy with a shape memory effect, which will make it possible to replace the more expensive alloys of this group and extend the scope of application. The studies allowed us to select the optimal chemical composition and to determine the best type of thermal treatment of the developed alloy. In particular, aging at a temperature of 800 °C for ten hours with subsequent cooling in the air enables obtaining a temporal resistance of the alloy to 1380 MPa.

It is demonstrated that the ageing under the second mode makes it possible to provide sufficient dispersion hardening to ensure operational properties at the high level.

The proposed iron-based alloy with a shape memory effect can be obtained both under laboratory conditions (the thin samples, the use of which in the industry is not feasible) and on industrial equipment, that is, to receive massive castings (parts).

The given alloy is expedient to apply for welding-free joining of structures, fuel supply lines, high-pressure pipelines, controlling elements in the form of couplings, springs, etc.

A positive aspect of the present study is obtaining mathematical dependences of the impact of each chemical element on tensile strength of the alloy and the values of a shape memory effect. However, an interesting point of further research would be finding the optimal values of each element and predicting the effect of chemical composition on operational properties of the alloy beyond experiment. In addition, of great practical importance for further research is adaptive modeling in the problem on optimal control of chemical composition in terms of impact on the operational properties of the alloy.

7. Conclusions

1. We examined a technological process for receiving an iron-based alloy with a shape memory effect, substantiated the choice of chemical composition of the alloy, selected thermal treatment modes. It was found that aging of the proposed alloy makes it possible to considerably improve the strength characteristics (up to 1380 MPa) and, at the same time, to increase the magnitude of SME (up to 97 %). It is demonstrated that in the proposed alloy the values of a shape memory effect, which was characterized by unbending angles of the samples, are quite high (125–160°).

2. It is shown that the proposed alloy possesses a high degree of shape recovery (78–97 %) while maintaining such important properties as strength (1180–1380 MPa), toughness, corrosion and scale resistance.

3. We constructed mathematical models in the form of dependences of the impact of chemical composition of the alloy on tensile strength of the alloy and the values of a shape memory effect. The models were received by using the second-degree polynomial approximation method with the verification of adequacy of the model by the magnitude of approximation credibility.

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