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INVESTIGATION OF PHYSICOMECHANICAL PROPERTIES AND TEMPERATURES OF TRANSFORMATION OF IRON-BASED ALLOYS

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***Abstract* – Investigations of powder alloys of the Fe- (30-36)% Ni system based on carbonyl powders are presented. When studying the microstructure of Fe-(30-36)%Ni alloys, it was found that an increase in the concentration of nickel promotes the coarsening of the average grain size of the austenite, and the grain coarsening promotes the acceleration of the austenitic - martensitic transformation. In alloys with nickel content in the range of 30-32%, the phase transformation temperature increased with increasing a minimum grain size of martensite in direct proportion to the nickel content. The critical austenite grain size was 1-3.5 microns. It is established that the transformation in alloys with 30.3 - 31.93% of nickel occurs in a wide range of temperatures (70-120 degrees), and in alloys with 34.35-36.33% of nickel - in a narrow one (1-2 degrees). It is shown that as the nickel content increases from 30 to 36%, the grain size increases and the temperature of the phase of γ-α transformation increases by 75 K. The fraction of martensite formed increases by a factor of 5. It was found that the decomposition of austenite in iron nickel alloys (30-32% Ni) was in the range of 70-92%, with a Ni content of 33-36%; the decomposition of austenite was insignificant: 2-15%. It was established that when the samples were cooled to 5 K in a field with a strength of 5 kE, the value of the magnetization increased to 113-189 emu / g. It was found that a decrease in the critical grain size provokes the transformation in alloys.**

Keywords– alloys, properties, phase, austenite, martensite, structure.

I. INTRODUCTION

Martensitic transformation (MT) is the most interesting stage of transformation of austenite in steels and alloys [1]. During the γ-α transformation process, martensite crystals are transformed to austenite grains over specific crystallographic planes and the interphase boundary does not appear in this case. At a given phase transformation, the oriented and batch shift of atoms in the austenite crystal lattice proceeds synchronously [2].

Superplasticity, superelasticity, shape memory effect and other abnormal mechanical properties of alloys are somehow connected with this transformation [3]. The notion of "martensitic transformation" emerged during the study of processes occurring during the rapid cooling of steels leading to a certain structure and high strength properties. In the second half of the twentieth century, the features of austenitic-martensitic transformations were formulated. Among these features, there is diffusion, shear character of the phase transformation, the rapid development of the transformation at low temperatures, the dependence of the martensite amount on temperature, and the specific "martensitic" structure [4].

To realize the martensitic transformation, first of all, it is necessary to subject the alloy to a rapid and continuous cooling below the starting point of the γ-α transformation (M_s) [5]. The lower the M_s, the higher the probability of the appearance of α-phase crystals. The amount of martensite that has arisen is growing as a result of the appearance of the newest crystals, and not as a result of the growth of existing crystals [6].

The temperature of the end of the transformation is denoted as M_f, the patterns of atomic rearrangement for γ-α transformations follow from certain crystal-geometric relationships between the crystal lattices (structures) of the γ and α-phases [7]. As a result of austenitic-martensitic transformation from one orientation of the austenite phase (grain, mono-crystal), 24 orientations of the martensitic phase can be obtained [8].

The amount of α phase, in comparison with austenitic, is dominated by the largest specific volume. In the manufacture of steels and alloys, it is necessary to observe arising α - phase [9]. Since an increase in martensite is the cause of the formation of internal stresses while carrying out heat treatment, these stresses in turn contribute to the appearance of deformation of the material and the formation of cracks in it.

To illustrate the description of all processes of γ-α transformations, three basic approaches can be used: crystallographic, thermodynamic approaches, and dynamic wave theory.

The crystallographic approach implies all the basic physical mechanisms of the austenite-martensitic transformation, for example, the deformation of austenite, which converts the original lattice of austenite into a martensite lattice [10].

All transformations of the martensitic type have a number of common features among themselves, such as the pattern of displacements of atoms relative to each other, the directivity of these displacements during the process of rearrangement of the crystal lattice of the initial phase into a new (forming) phase and the cooperativity of atomic displacements. These features lead to macroscopic displacement and the appearance of relief on the surface. The results of G.V. Kurdyumov lead to the determination of martensitic transformation - "Martensitic transformation consists in the regular restructuring of the lattice, in which atoms do not exchange with places, but only shift relative to each other over distances not exceeding interatomic".

The thermodynamic approach implies that under the conditions of supercooling, a diffusion decomposition of austenite into a ferritocarbide mixture can not occur, which brings the system to an absolute minimum of free energy. [9].

In alloys with the lowest martensitic point, the transformation during cooling is a series of small explosions, and there is no increase in the amount of martensite at a constant temperature. Lowering the temperature does not slow down this transformation and increase the cooling rate accordingly, it does not allow delaying the formation of martensite.

The dynamic wave theory of the γ-α transformation is based on the concept of heterogeneous nucleation and controlled wave growth. The theory implies that the transformation does not begin at the grain boundary, but occurs at some distance from it, namely in the volume of the grain. The point of onset of the nucleation of α-phase crystals is dependent on the propagation along the grain of elastic deformations, which are caused by the presence of defects-dislocations. Grain, having a minimum size, in which dislocations can not fit, in turn will not support the phase transformation [11].

To date, many works have been published both for interpreting the features of austenitic-martensitic transformation, related to the carriers of threshold deformation, and for creating a dynamic approach to the theory of the formation of martensite crystals. The idea of a theory additional to the thermodynamic approach is reduced to deciphering the dynamic structure of the excited state of the lattice in the nonequilibrium front region of the nonlinear transformation wave [12]. One of the scenarios for the growth of martensite from the position of the wave theory indicates that in the regions adjacent to the boundary, when a martensitic crystal interacts with a boundary, dislocations may be generated, which become the centers of nucleation of a new martensitic crystal [13].

All the theories and approaches to the description of martensitic transformations that exist today can be applied depending on the composition of the material and the technologies for its production [14]. The aim of this work is to study the physico-mechanical properties and temperatures of phase transformations of alloys of the Fe - Ni system [15].

II. METHODS

To produce samples of powder alloys of the Fe-Ni system, the authors used carbonyl iron powders - grade VMS-1, carbonyl nickel - grade PNK UT-3. The powders were mixed for 8 hours in a mixer, with a shifted axis of rotation. The blanks were pressed in a mold on a hydraulic press P-125, at a pressure of 600 MPa.

The sintering of the samples was carried out in a vacuum electric furnace SSHV-4.5.5 / 12-IC1. Annealing was performed at t° = 900ºC for 1 hour. The samples were press at 600 MPa; the cycle was repeated twice. Sintering was carried out in vacuum and argon media at t ° = 1200 ° C, in total, for 20 hours [16].

To detect the microstructure (austenite grain and martensitic phase), used thermal etching of the sample surface in a vacuum medium at t°=1000°C was used for 30 minutes. The microstructure images were obtained through an optical microscope Axiovert and an analytical, scanning, electron microscope ULTRA 55/60 Carl Zaiss, with a resolution of 1 nm, equipped with an INCA spectral analyzer [17].

The microhardness of the austenite and martensitic phases was measured with PMT-3, at a load of 0.5 N, in accordance with GOST 9450-76, the measurement error was not more than 10%. The grain sizes were calculated by measuring the length of chords in accordance with GOST 5639-82 on microstructure images with a magnification of 200 times, as well as using specialized software (Gwydion SoftWare). X-ray phase analysis of samples of powder alloys was obtained using a Shimazu XRD 6000 diffractometer in K-α-Cu radiation. Decoding the diffractograms was carried out using the ICDD PDF-2 map file. The hardness of the powder alloys after sintering was measured using the Brinell method [18].

The tensile strength and Young's modulus were determined on INSTRON 5885 [19]. The impact strength was determined in accordance with GOST - 9454-78 at room and negative (-190 ° C) temperatures. The magnetometric method was used to study phase γ-α transformations in powder alloys of the Fe- (30-36)% Ni system. Samples of alloys after sintering were magnetized in an increasing magnetic field: from 0 to 5 kE, at room temperature, after which the samples of the alloys were supercooled to 5 K in a field with a strength of 5 kE. The magnetization of the samples increased to 113-189 emu/g, because of the appearance of additional magnetization of the α phase, which in turn was formed as a result of supercooling of the alloys.

III. DISCUSSION AND RESULTS

X-ray phase analysis showed that an austenite structure was formed in all the alloys after sintering. The grain structure of sintered alloys Fe-30% Ni and Fe-36% Ni is shown in Fig. 1 a, b. The structure is characterized by heterogeneity, pores are visible in the structure, twins are present in the grains, which indicates the presence of austenite. The histograms of the grain size distribution are shown in Fig. 1c, d.

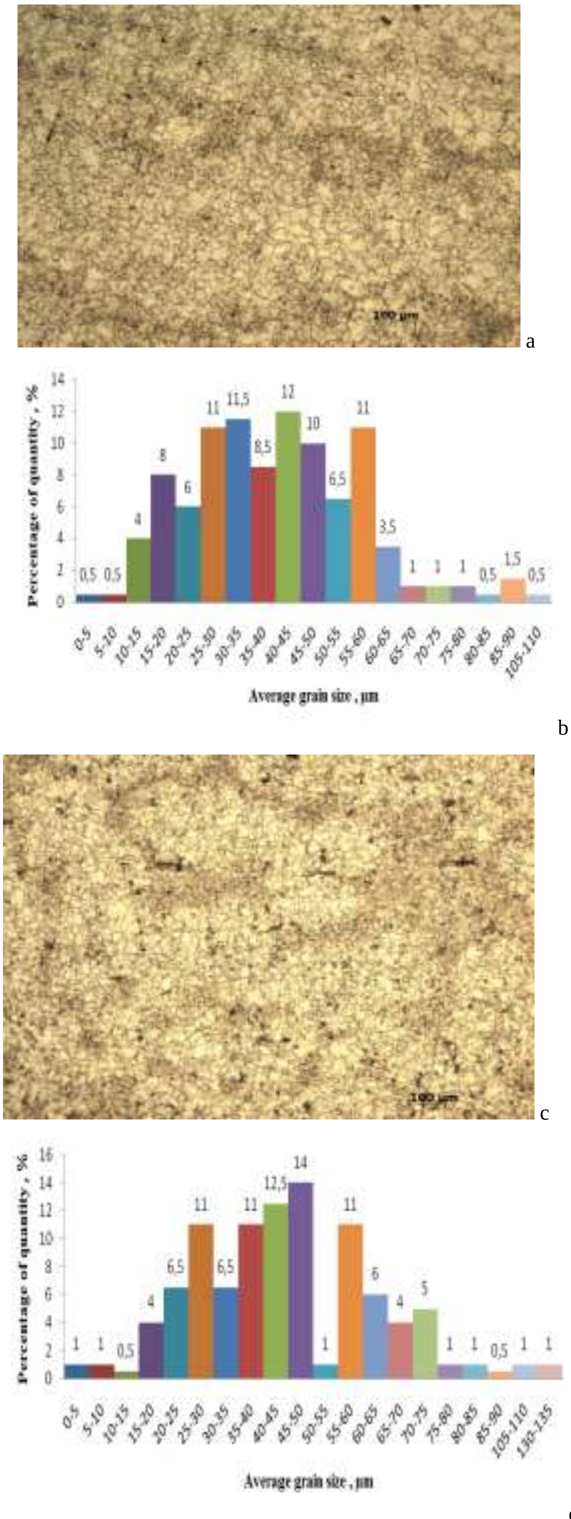


Fig. 1. a, b: photographs of the structure of Fe-30% Ni and Fe-36% Ni alloys, respectively, in g: histogram of grain size distribution in Fe-30% Ni and Fe-36% Ni alloys

TABLE 1. Physical and mechanical properties and grain size of Fe- (30-36)% Ni samples after sintering

№	Ni content,%	Microhardness γ, MPa	P, %	KC, kJ/m^2	Average grain size , μm
2	30.2	1610	5	807	33±15
4	30.5	1450	4	810	45±15
6	31.9	1452	2	817	39±13
8	31.6	1668	4	815	41±13
20	33.1	1478	5	829	39±9
30	34.3	1714	6	868	45±16
38	36.3	1620	6	880	52±16

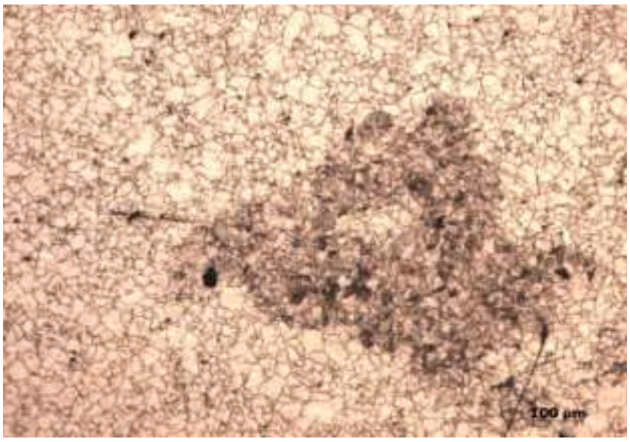
After sintering the samples of powder alloys of the Fe-(30-36,%) Ni system, the residual porosity of the samples was 2-6%, Table 1.

The grain size in Fe Ni alloys increased,insignificantly wit h increasing nickel concentration: from 30 to 50 μm. It is established that an increase in the nickel content in alloys contributes to an increase in the toughness from 807 to 880 kJ / m^2. The microhardness of the austenite after sintering was 1450-1714 MPa, in proportion to the nickel content.

After cooling the alloys to -190 ° C (cooling in liquid nitrogen) in iron-nickel samples with a Ni content of 33 to 36%, the decomposition of austenite was insignificant (2-15%), Table. 2, in the structure after cooling, two phases are distinguishable: austenite and acicular martensite, Fig. 2

TABLE 2. Phase composition of the samples after cooling of the Fe- (30.2-36.3)% Ni powder system

№	Ni,%	Y-phase,%	α-phase, %	Microhardness after cooling, MPa	
				A	M
4	30.2	8	92	2110	2370
8	31.6	30	70	1880	2440
6	31.9	15	85	1940	2600
20	33.1	85	15	2230	2800
38	35.8	97	3	2300	3580
28	36.3	98	2	2370	3810



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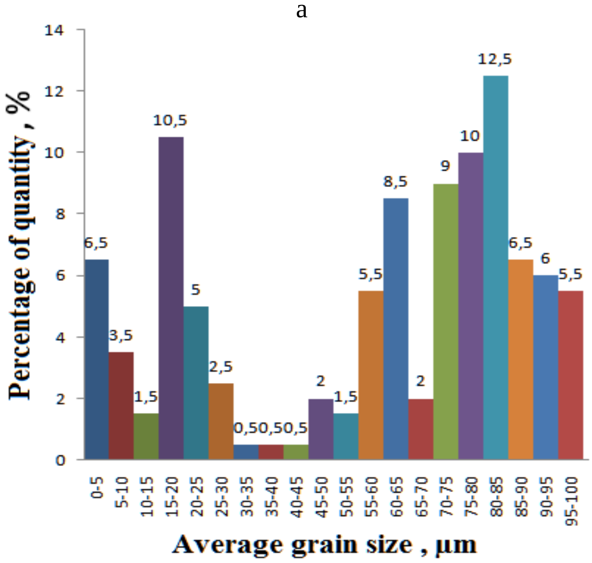

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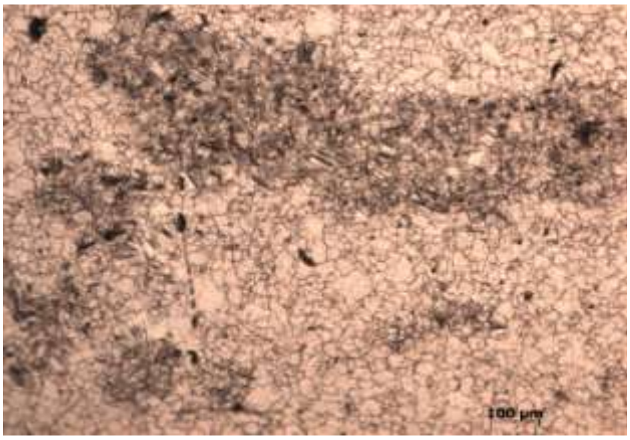

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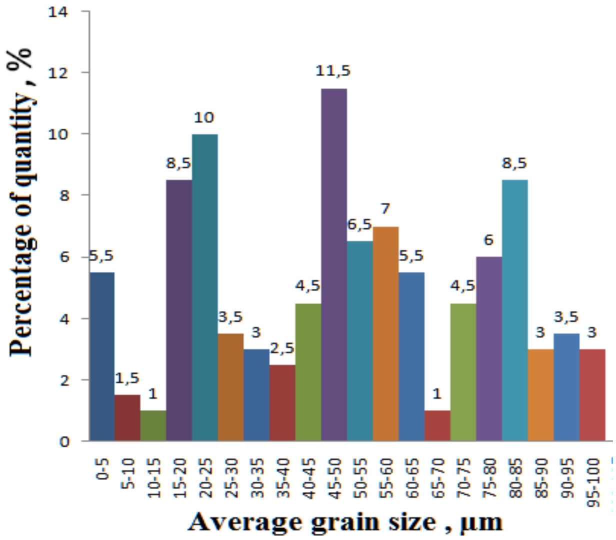


d

Fig. 2. a, b: photographs of the structure of Fe-30.296% Ni and Fe-36.33% Ni alloys after cooling, c, d: histograms of the distribution of austenite grains in size in alloys Fe-30.296% Ni and Fe-36.33% Ni

After supercooling the samples, the microhardness of the austenite phase was measured, which was 1880-2370 MPa, as well as the resulting martensitic phase - 2370-3810 MPa, (Table 2), depending on the nickel content. Testing the toughness of alloys at -190 ° C showed an increase in this strength characteristic to 350 kJ / m^2, with an increase in the nickel content from 30 to 36%, Fig. 3.

The hardness decreased insignificantly with increasing nickel content, apparently, the increase in porosity had an effect on the decrease in hardness, Fig 4. The microhardness of the alloys increased in proportion to the concentration of nickel, Fig. 5.

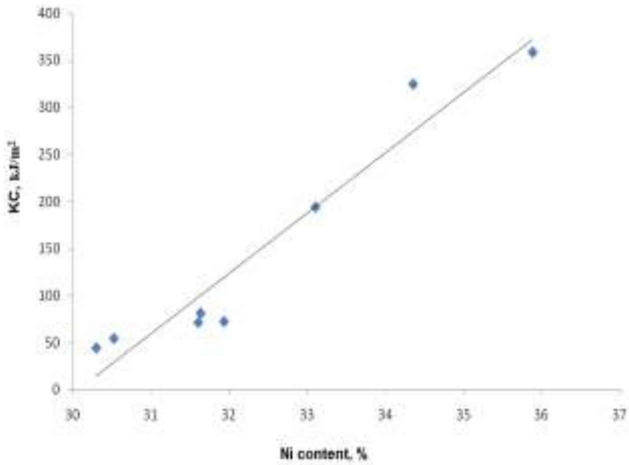


Fig. 3. Dependence of the toughness of samples of the Fe- (30-36)% Ni system on the Ni content at -190°C

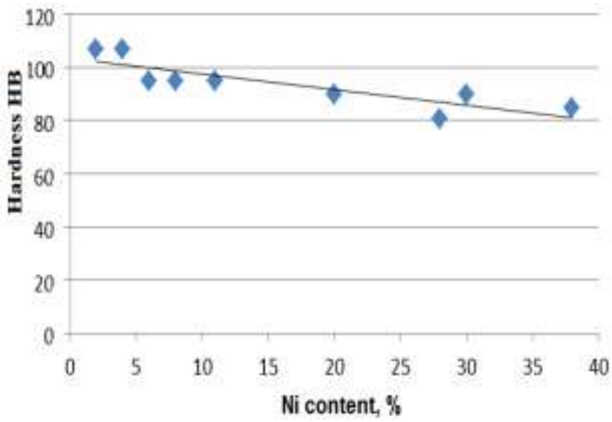


Fig. 4. Dependence of the hardness of samples of the Fe- (30-36)% Ni system on the Ni content

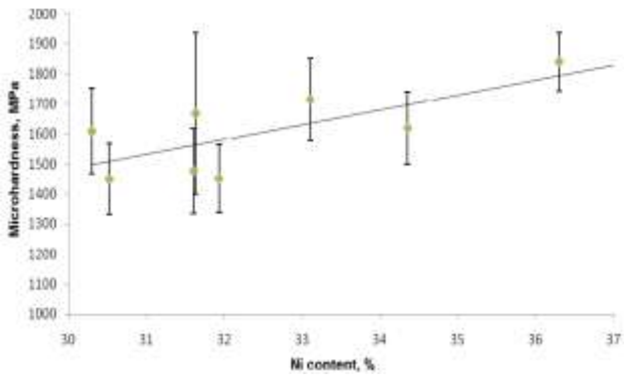


Fig. 5. Dependence of the microhardness of samples of the Fe- (30-36)% Ni system on the Ni content

Young's modulus of iron-nickel alloys increased from 4700 to 5500 MPa, with an increase in the nickel content from 30 to 36%, Fig. 6, the tensile strength index decreased to an increase in the nickel concentration, the yield strength increased with increasing Ni content, Fig. 7. In Fe- (30-36)% Ni alloys, the temperatures of the beginning and the end of the γ-α transformation were determined by a magnetometric method (Table 3, Fig. 8).

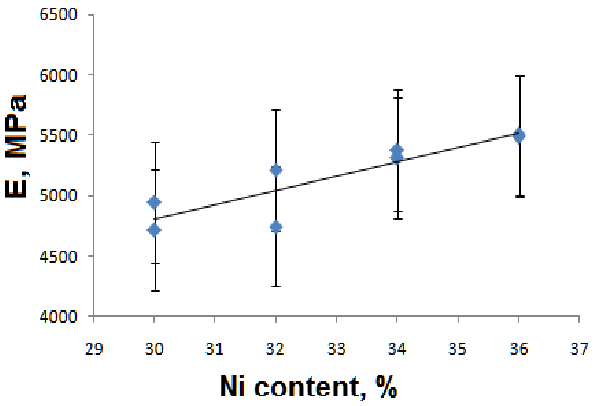


Fig. 6. Dependence of Young's modulus (E) of samples of the Fe- (30-36)% Ni system on the change in the Ni content in alloys

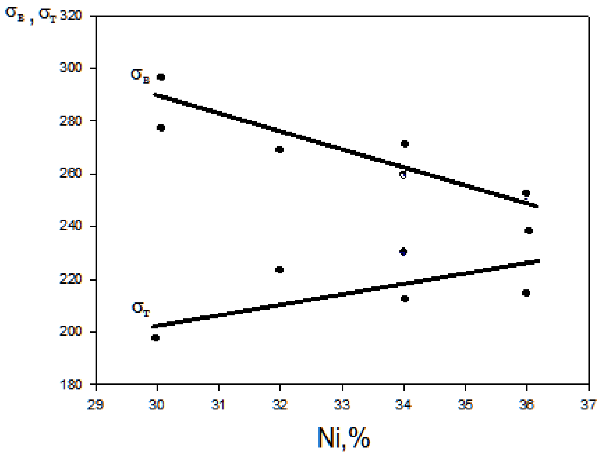


Fig. 7. Graphs of the ultimate strength and yield strength of the powder system samples: Fe- (30-36)% Ni, depending on the concentration of nickel

TABLE 3. The temperatures of the beginning (T2) and the termination (T1) of the austenitic-martensitic transformation

Ni content.%	T_1, K	T_2, K	M_{st} (emu/g)	M_{fin} (emu/g)	d_{cr}, µm
30.3	58.2	169.2	43.1	189.5	1
31.85	70.2	142	70.2	185.7	-
31.93	55.2	173.3	71.6	185.4	3,5
34.35	109.2	111	113.8	177.9	-
36.33	134.2	135.5	148.3	170.4	-

Transformation in alloys with 30.30 - 31.93% nickel occurs in a wide range of temperatures (70-120 degrees), and in alloys with 34.35-36.33% nickel - in a narrow (1-2 degrees), Fig. 8.

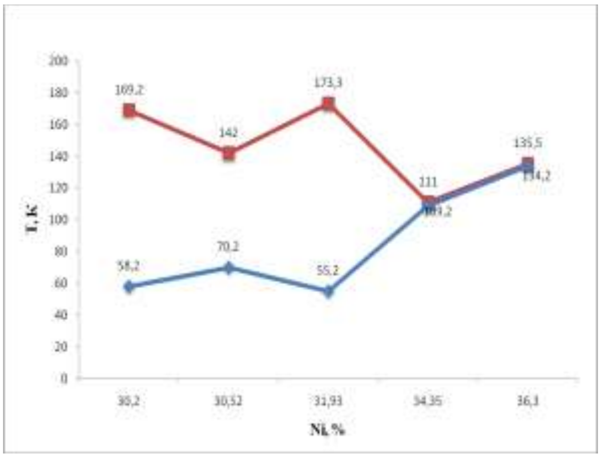
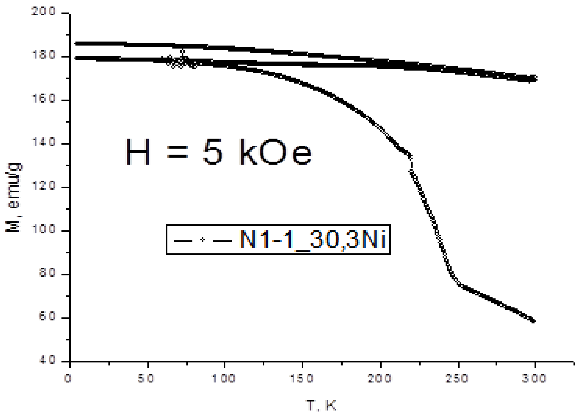


Fig. 8. Start temperature (upper graph) and finish temperature (lower graph) of austenitic-martensitic transformation in Fe- (30.2-36.3)% Ni alloys



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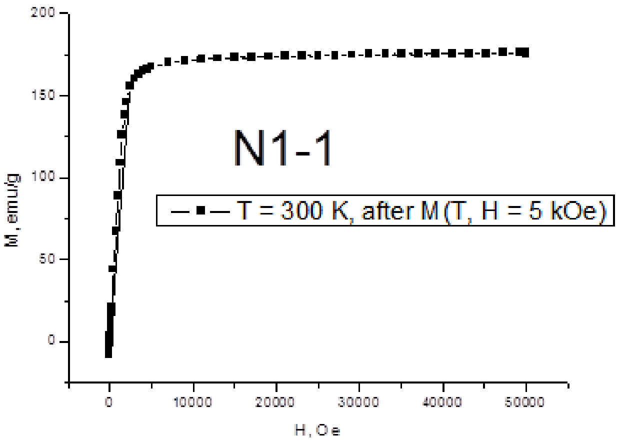
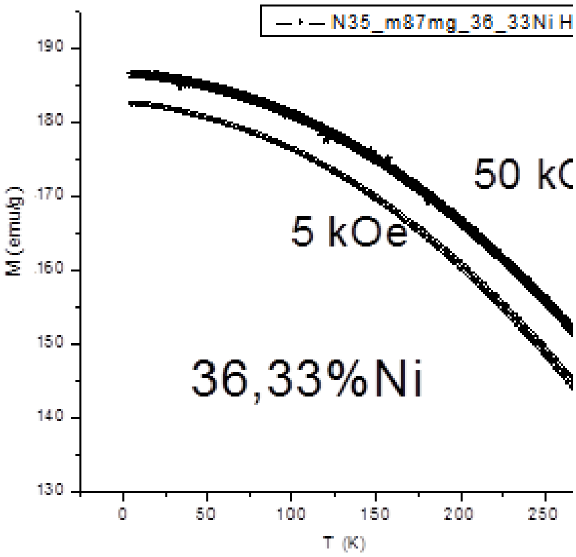


b

Fig. 9. Graphs of the magnetization of the alloy by the temperature (a), the field dependence of the magnetization at a constant T = 300 K (b) sample Fe-30.29% Ni



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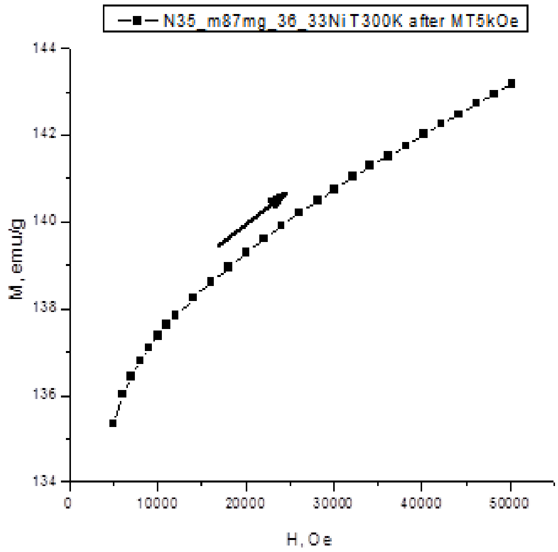

b

Fig. 10. Graphs of the magnetization of the alloy by the temperature (a), the field dependence of the magnetization at a constant T = 300 K (b) sample Fe-36.33% Ni

This is due to the fact that in alloys with an increased nickel content, the transformation occurred in the grains of a narrow size range (1-30 μm), and in the alloys with a reduced content, grains with sizes from 1 μm to 60 μm were tested. Probably, a certain temperature of the phase transformation, which agrees with the dynamic wave theory of austenite-martensitic transformation, corresponds to each size range.

In the process of cooling the samples to 5 K, in a field with a strength of 5 kE, the value of the magnetization increased to 113-183 emu / g (Fig. 9-10), because an additional magnetization appeared from the appearance of martensite. The grain size distribution histograms (with maximum grain sizes up to 100 μm) shows that for the Fe-Ni alloys with concentrations of 30.3% and 31.93%, the critical Dc size is close to 1 μm and 3.5 μm, respectively.

The transition to concentrations of 34.35% and 35.33% is not accompanied by the appearance of a significant amount of martensite, an increase in the concentration of nickel leads to an increase in the temperature of the onset of conversion and a decrease in the temperature of the end of the transformation. Parameter Dc is not a constant within the framework of the microscopic theory.

When approaching 34% of nickel, Dc tends to infinity; initial point Ms tends to zero, and transformation does not occur independently of the single crystal. After the inclusion of a strong magnetic field, Dc significantly decreases - the transformation continues, the temperature of Ms increases - a transformation that did not initially exist appeared. This shows that already about 34.35% concentration is near critical value C *, and the values of Dc (in accordance with the known histograms) are not less than 90 μm. This result is of fundamental character and agrees with the conclusions of the dynamic theory of martensitic transformations.

IV. CONCLUSION

When studying the powder system Fe- (30-36)% Ni alloys, it was found that as the nickel concentration increases, the hardness is reduced to 20%, and the yield strength and the elastic modulus (Young) increase to 20%. The microhardness of austenite and martensite after their cooling in liquid nitrogen increased with increasing nickel concentration. Tests on the toughness of alloys at negative temperatures (-190 ° C) showed an increase in this index to 350 kJ / m^2 with an increase in the Ni content from 30.2 to 36.3%.

With decreasing nickel concentration, an increase in the temperature range of the austenite-martensitic transformation is associated with a broad grain diameter and there is an increase in the γ-α phase transition temperature from the critical grain size, which fits within the framework of the Dynamic Wave Transformation Theory.

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