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Research Article

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Diffusion Processes in the Formation of the Structure of Alloyed Powder Steels

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Abstract

Introduction. The production of alloyed powder steels continues to be one of the most promising areas in domestic powder metallurgy. This is due to the high level of performance characteristics and the wide range of products that can be produced. Creating materials with desired properties is a complex process that involves various phenomena. One of these phenomena is the diffusion alloying of iron-based powder steels, which plays a special role in this process. The creation of alloyed powder steels in the Fe-NiO and Fe-Ni systems is important for metallurgy and metalworking, as they are used for coating and sintering to obtain materials with specific properties. In addition, the diffusion of nickel in iron during heat treatment is considered to improve material properties. Recent advances in the study of mutual diffusion are associated with the investigation of homogeneous systems. However, mutual diffusion even in single crystals always occurs under spatially inhomogeneous conditions. The modern literature has not sufficiently studied the mutual diffusion in two- and multi-component powder systems. Therefore, the aim of this work is to determine the effect of diffusion alloying with nickel and nickel oxide of iron-based powder steel on the processes of obtaining powder materials. Within the framework of this goal, the following tasks were set: to investigate the diffusion processes of interactions between pairs in the Fe-NiO and Fe-Ni systems, as well as to study technological modes of sintering and reducing annealing of samples in order to achieve maximum mechanical properties that would ensure the formation of a high-quality product.

Materials and Methods. The work used iron powder of the PZHRV 2.200.26 brand manufactured by PJSC Severstal (Cherepovet) and carbonyl nickel powder PNK-UT3, obtained by the electrolytic method or splitting nickel salt with an aqueous solution, according to GOST 97922–97. Before use, the powders were tested using a universal laser particle size measuring device model FRITSCH ANALYSETTE 22 MicroTecplus and a Beckman COULTER No. 5 submicron particle analyzer. A two-cone mixer RT-NM05S (Taiwan) was used to prepare the charge. Pressing was carried out on a hydraulic press model TS0500–6 (China) in laboratory molds. Samples were obtained by pressing pre-hardened 3 mm diameter powder pins into a carbonyl nickel or NiO charge with a dispersion of 5–10 microns. Recovery annealing was carried out in a SNOL 6.7/1300 laboratory muffle furnace at 700°C, followed by annealing-sintering at temperatures of 1,050, 1,150 and 1,250°C in a hydrogen atmosphere for 9 hours.

Microstructural analysis was performed using a NEOPHOT-21 optical microscope. A Hitachi S-3400N scanning electron microscope was used to study the fine structure of the material. The distribution of element concentrations in the Fe-Ni diffusion zone was studied by local X-ray spectral analysis using the Kamebaks installation.

Results. The studies showed that the porosity of the powder component after pressing was 12%. Diffusion in the iron-nickel powder system was 5–10 times higher when using carbonyl nickel compared to oxide. It was also found that high diffusion rates of reduced nickel led to faster and more uniform penetration of alloying elements into the material. The dependences of the distribution of nickel concentration and its oxide content after sintering were

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determined, as well as the indicators of diffusion interaction between iron, nickel, and nickel oxide during annealing, where nickel oxide was reduced and sintering occurred at different temperatures.

Discussion and Conclusion. The analysis of the results obtained indicates a different intensity of diffusion processes in powder-alloyed steels. This can be explained by both the distortion of the crystal lattice of the starting materials and the increased segregation of defects, such as defective zones, that are formed during compaction of the material. This approach to studying two-component diffusion allowed us to compare the intensity of element diffusion redistribution depending on chemical composition and temperature, and to estimate the effective activation energy of diffusion. As a result of our studies, we have established quantitative parameters for the distribution of nickel concentration in the iron matrix, depending on sintering temperature, which affects the formation of high-quality materials. The research results obtained are of interest to specialists in powder metallurgy and heat treatment, as they can be used in the development of new multicomponent alloys.

Keywords: powder steels, nickel oxide, nickel, diffusion, structure formation, sintering, splicing, contact section, mechanical properties

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Научная статья

Диффузионные процессы при формировании структуры легированных порошковых сталей

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Аннотация

Введение. Производство легированных порошковых сталей остается одним из перспективных направлений отечественной порошковой металлургии, что объясняется высоким уровнем эксплуатационных свойств и широкой номенклатурой получаемых изделий. Формирование таких материалов с требуемыми свойствами является сложным процессом, сопровождающимся многообразными явлениями, особое место среди которых занимает диффузионное легирование железной основы порошковых сталей. Создание легированных порошковых сталей в системе Fe-NiO и Fe-Ni имеет важное значение в металлургии и металлообработке при процессах нанесения покрытий и спекания для получения материалов с требуемыми свойствами. В процессе термической обработки для улучшения свойств материалов рассматривается и диффузионное взаимодействие никеля в железе. В последнее время успехи в изучении взаимной диффузии связаны с исследованием однородных систем. Однако взаимная диффузия даже в монокристаллах всегда протекает в пространственно-неоднородных условиях. В современной литературе недостаточно исследована взаимная диффузия в двух- и многокомпонентных порошковых системах. Поэтому целью работы является определение влияния диффузионного легирования никелем и оксидом никеля порошковой стали на основе железа на процессы получения порошковых материалов. В рамках обозначенной цели поставили задачи — исследовать диффузионные процессы взаимодействия пар в системе Fe-NiO и Fe-Ni, технологические режимы спекания и восстановительный отжиг образцов для достижения максимальных механических характеристик, которые бы обеспечивали формирование качественного материала.

Материалы и методы. В работе использовался железный порошок марки ПЖРВ 2.200.26 производства ПАО «Северсталь» (г. Череповец) и карбонильный порошок никелевый ПНК-УТ3 ГОСТ 97922–97, получаемый электролитическим методом или путем расщепления никелевой соли водным раствором. Перед использованием порошки проходили контроль на универсальном лазерном приборе измерения размера частиц модели FRITSCH ANALYSETTE 22 MicroTecplus и анализаторе субмикронных частиц Beckman COULTER №5.

Для приготовления шихты использовали двухконусный смеситель марки RT-NM05S (Тайвань). Прессование осуществлялось на гидравлическом прессе модели TS0500-6 (Китай) в лабораторных пресс-формах. Образцы получали запрессовкой заранее упрочненного порошкового штифта ø 3 мм в шихту карбонильного никеля или NiO с дисперсностью 5–10 мкм. Восстановительный отжиг образцов осуществляли в муфельной лабораторной печи SNOL 6,7/1300 при температуре 700 °C и отжиг-спекание при 1 050, 1 150, 1 250 °C в атмосфере водорода в течение 9 часов.

Фиксирование микроструктуры выполнялось на оптическом микроскопе «NEOPHOT-21». Тонкое строение структуры изучали на сканирующем электронном микроскопе Hitachi S-3400N. Распределение концентрации элементов в диффузионной зоне Fe-Ni изучали методом локального рентгеноспектрального анализа на установке «Камебакс».

Результаты исследования. Пористость порошкового компонента после прессования составляла 12 %. Диффузия в системе железо-порошок никеля, восстановленном из оксида, в 5–10 раз выше, чем при использовании порошка карбонильного никеля. Установлено, что высокая скорость диффузии восстановленного никеля приводит к более быстрому и равномерному проникновению легирующих элементов в материал. Определены зависимости распределения концентрации никеля и его оксида после спекания, а также рассчитаны показатели диффузионного взаимодействия между железом, никелем и оксидом никеля после операции отжига, при котором происходит восстановление оксида никеля и спекание при различных температурах.

Обсуждение и заключение. Анализ полученных результатов свидетельствует о различной интенсивности диффузионных процессов в порошковых легированных сталях. Объясняется это как искаженностью кристаллической решетки исходных материалов, так и увеличенной сегрегацией дефектов (содержанием дефектных зон), которые образуются в процессе уплотнения материала. Данный подход к исследованию двухкомпонентной диффузии позволил сопоставить интенсивности диффузионного перераспределения элементов в зависимости от химического состава и температуры, а также оценить эффективную энергию активации диффузии. В результате выполненных исследований установлены количественные параметры распределения концентрации Ni в железную основу в зависимости от температуры спекания, влияющие на формирование качественного материала. Полученные результаты исследований интересны специалистам в области порошковой металлургии и термической обработки при разработке новых многокомпонентных сплавов.

Ключевые слова: порошковые стали, оксид никеля, никель, диффузия, структрообразование, спекание, сращивание, контактное сечение, механические свойства

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Introduction. The performance characteristics of the material, both in its compact and powder forms, are primarily determined by its chemical composition and structural state [1, 2]. Depending on the purpose, specific requirements are imposed on the microstructure [3]. The formation of a specific structure of powder steel during sintering also depends on the processes that occur during sintering and subsequent heat treatment [4]. Many factors influence the process of structure formation in powder materials [5]. According to [6], the main factors are the methods of their production, granulometric composition, compacting pressure, sintering medium and time, temperature of deformation action. The introduction of alloying elements that affect the processes in the surface layers of particles during solid-phase sintering, and additives forming the liquid phase, is the most effective way to increase the level of operational properties [7]. Each of the factors has its advantages and disadvantages.

Powder alloyed steels have a high level of performance properties. They are characterized by a homogeneous structure and a homogeneous distribution of alloying elements throughout the steel structure [8]. When impregnated with liquid metals, for example, when using the cementation or carbonitration process, the alloying elements are absorbed by the surface layers of the material. This may lead to the formation of a surface layer with a high concentration of alloying elements, but deeper layers may be less saturated. Therefore, in such cases, impregnation with liquid metals is most effective for creating a surface layer with the desired properties [9]. Diffusion saturation can also lead to an uneven distribution of alloying elements. Diffusion can be limited only by surface layers of the material or internal defects, which limits the uniformity of saturation over the entire volume of the part. The choice of alloying method depends on the purpose, required properties, size and shape of the part, available resources and other factors. It is important to carefully consider all these factors when selecting a method to ensure optimal results.

The method of introducing nickel into powder structural steels in the form of additives of dispersed nickel oxides has proven itself well [10]. The recovery annealing of an iron-based charge with NiO particles evenly distributed in it leads to the recovery and fixation of the reduced nickel particles on iron particles by surface and grain boundary diffusion. The use of such a partially alloyed powder makes it possible to obtain parts with a homogeneous structure and a high complex of mechanical properties at a lower temperature. Activation of homogenization process of powder steels using oxides of alloying elements in the literature is explained only at a qualitative level [11]. Therefore, the aim of this work was to study the processes of diffusion interaction in Fe-Ni and Fe-NiO systems to determine the quantitative parameters of diffusion in them. The main task within the framework of this goal was to study the influence of technological modes of sintering and diffusion annealing during diffusion interaction in a system of Fe-Ni and Fe-NiO pairs on the formation of high-quality powder steel.

Materials and Methods. In the work, powder PZHRV 2.200.26, produced by Severstal PJSC (Cherepovets) and nickel carbonyl powder PNK-UT3 GOST 97922-97, obtained by electrolytic method or by splitting nickel salt with an aqueous solution, were used [12]. Data on the total chemical composition are presented in Table 1.

Table 1

Chemical composition of PZHRV 2.200.26 powder

Powder grade	Mass content of components, %									
	Mo	Ni	C	O	H	Cu	Si	Mn	P	S
PZHRV 2.200.26	–	–	0.090	0.140	–	–	0.014	0.087	0.012	0.005

Technological properties of PZHRV 2.200.26 powder: bulk density — from 2.4 to 3.0 g/cm^3; fluidity — no more than 37 sec/50 g; density at P=700 MPa — 7.0–7.05 g/cm^3; strength at a density of 6.5 g/cm^3 — more than 14 N/mm^2. The analog is powders from the Swedish company Höganäs: AHC 100.29, NC 100.24, SC 100.26, ASC100.29. Scope of application: shock absorption group parts, transmission parts, body parts, gears, connecting rods, couplings, bushings, etc. Main qualities of PNK-UT3 nickel powder include high corrosion resistance and resistance to aggressive environments.

Powder compositions are characterized by a high intensity of boundary and surface processes, which, during diffusion, can lead to an intensification of mass transfer [13]. The regularity of the structure formation of powder materials has been studied. The processes of diffusion interaction of alloying components were modeled using the diffusion pair method [14].

Granulometric composition of PZHRV 2.200.26 iron powder was determined using a universal laser particle size measuring device of a FRITSCH ANALYSETTE 22 MicroTecplus model (Germany) and a Beckman COULTER submicron particle analyzer No. 5 (USA).

Diffusion processes were considered on model pairs of powders of the grades PZHRV 2.200.26 (powder) — Ni (carbonyl); PZHRV 2.200.26 (powder) – NiO, which were manufactured by pressing a rod from the PZHRV 2.200.26 material with a diameter of 3 mm into a powder of carbonyl Ni or NiO with a dispersion of 5–10 microns. Recovery annealing of steam samples was performed at a temperature of 700°C and annealing-sintering at 1,050°C, 1,150°C, and 1,250°C in a hydrogen atmosphere for 9 hours [15].

Fe-Ni pair refers to systems with unlimited solubility of components. Iron and nickel at the temperature of diffusion annealing possess FCC lattices with similar parameters and form solid substitution solutions. Upon cooling, iron alloys containing less than 6% of nickel recrystallize into a ferritic phase with a BCC lattice. Metallographic studies were carried out after annealing with preliminary preparation of the sections in a plane perpendicular to the initial boundary between the components of the pair.

The distribution of concentration of elements in the Fe-Ni diffusion zone was studied by local X-ray spectral analysis at a Camebax installation manufactured by Cameca (France). The survey was carried out in the plane of the section in the direction perpendicular to the initial boundary between the components of the pair (along the axis of the primary cylindrical iron sample). Four concentration curves of distribution of relative intensities of K_{a1} line of iron and nickel were recorded from each section along the cross section of the diffusion zone in increments of 1 μm. The nickel and iron contents were determined at each point of the concentration curve under study. The formation of the diffusion zone increased 1.6–2.2 times due to the presence of nickel oxide paired with iron compared to pure nickel [16]. The coefficients of mutual diffusion $\overline{D}$ during sintering in the ranges of 1,050°C–1,250°C were determined depending on the content of alloying elements [17, 18].

Microstructure was fixed using an optical microscope “NEOPHOT–21” by Carl Zeiss Jena (Germany). The fine structure of the sample was studied using a Hitachi S-3400N scanning electron microscope (Taiwan).

The calculations were performed using mathematical methods based on the Matano method [16].

Results. The porosity of the powder component after pressing was 12%. Granulometric composition of PZHRV 2.200.26 iron powder was determined on the histogram: there was a single peak indicating that the powder was monofractive (Fig. 1). The average particle size was 98.5 microns. The results are presented in Table 2.

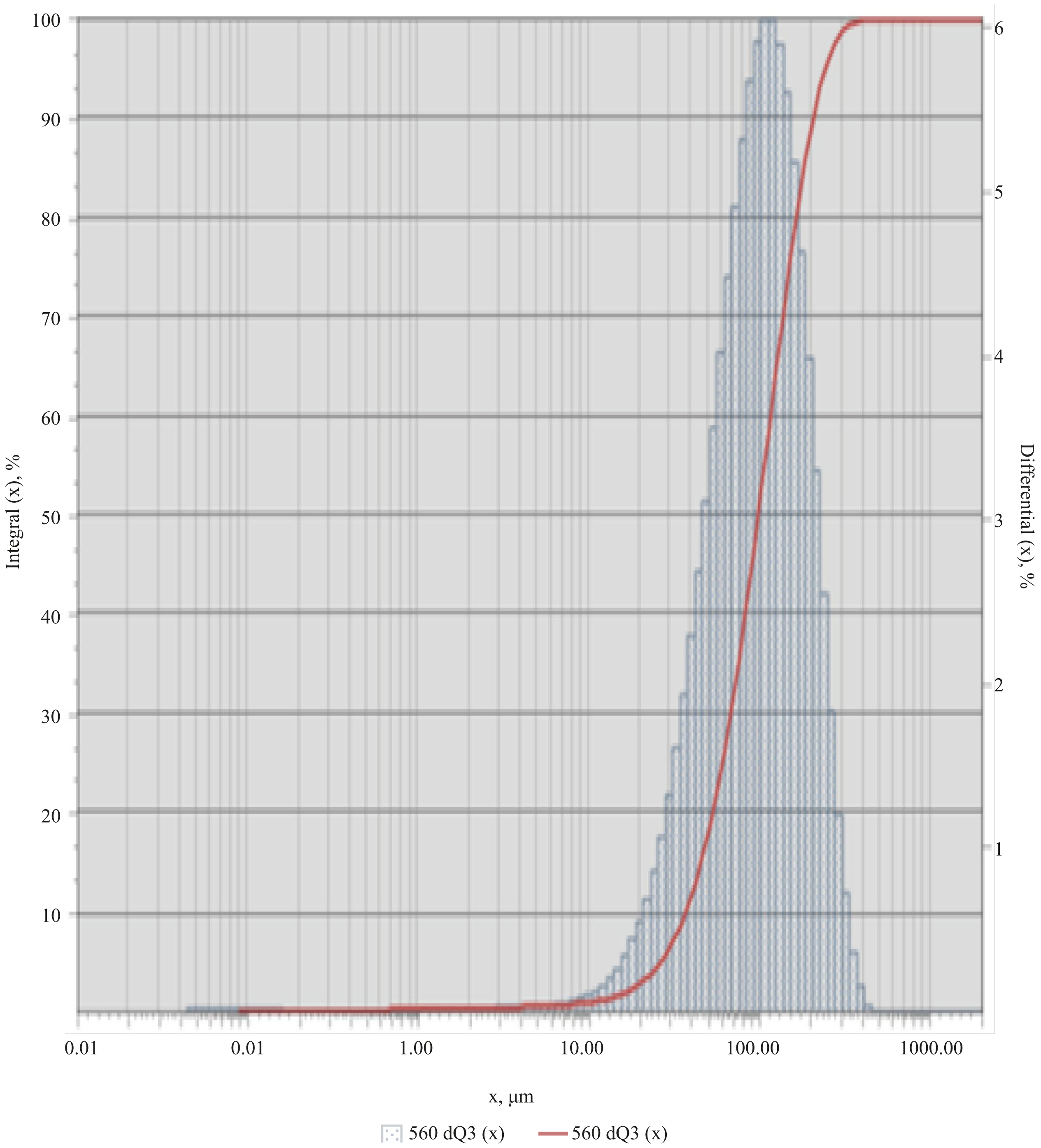


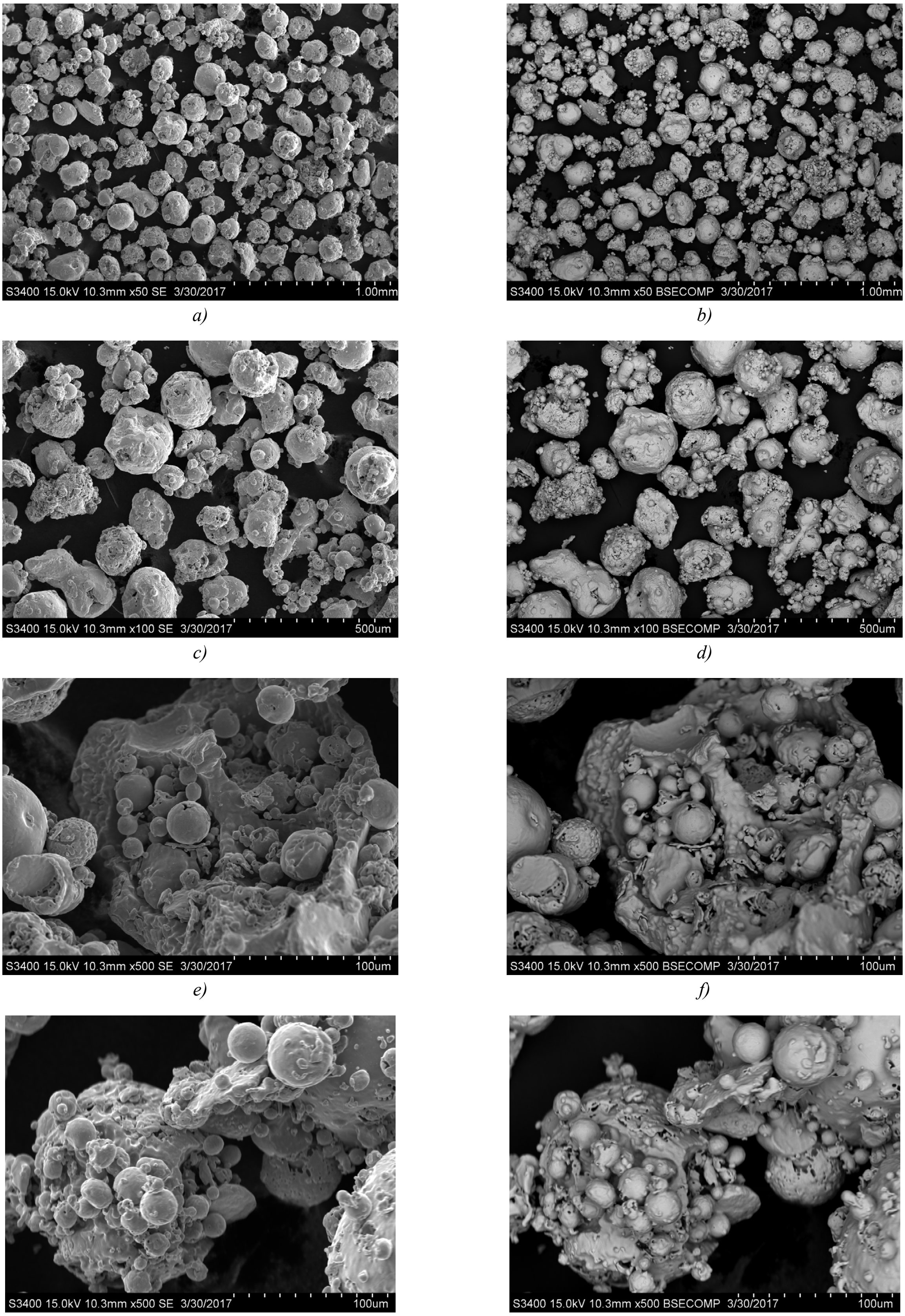
Fig. 1. Granulometric composition of PZHRV 2.200.26 iron powder

Table 2

Granulometric composition of PZHRV 2.200.26 iron powder

Quantitative share, %	5	10	25	50	75	90	95	99
Dimension, microns	26.5	37.0	60.7	98.5	149.8	206.4	244.0	317.4

Figures 2 *a* and *b* present a general view of the iron powder of the PZHRV 2.200.28 grade. Figures 2 *c, d, e, f, g, h* show the morphology of iron powder particles obtained using a Hitachi S-3400N scanning electron microscope.



a) *b)*

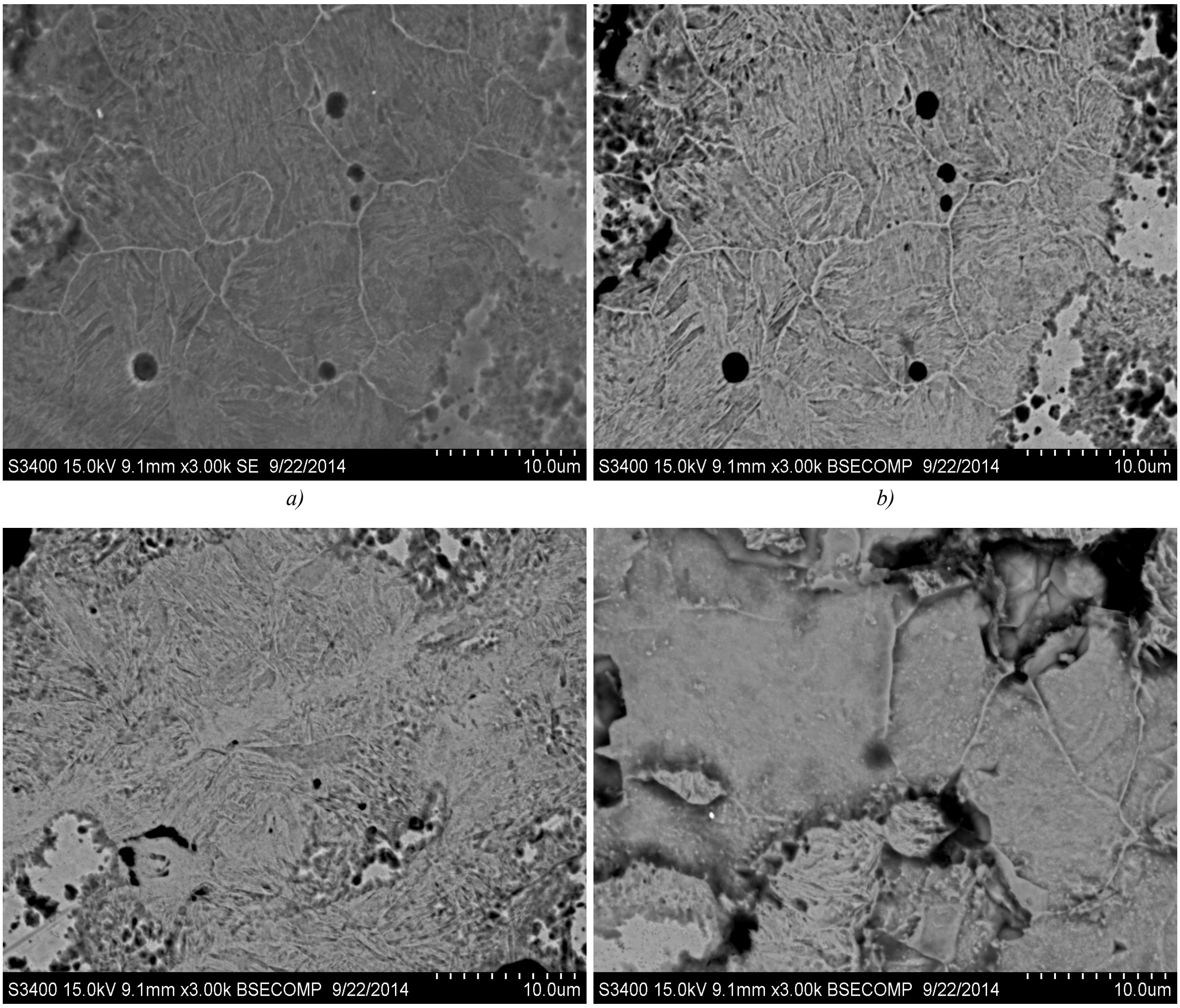
c) *d)*

e) *f)*

g) *h)*

Fig. 2. SEM image of PZHRV 2.200.28 iron powder shooting in:
a, c, e, g — reflected electrons; *b, d, f, h* — in secondary electrons

The structure of the diffusion zones consisted of ferritic and austenitic phases separated by a boundary. At a sintering temperature of 1,050°C, a porous austenitic phase was observed (especially in samples containing NiO oxide), due to high negative volumetric effects during the recovery of NiO. Sintering led to more intensive compaction when the temperature rose to 1,150°C and 1,250°C. Intensification was observed in samples reduced from Ni oxide. A significant increase in the volumetric fluidity of the material after the recovery of nickel oxide particles was explained by increased structural unevenness and incorrect arrangement of dislocations, in comparison with the structure of a powder material with nickel particles. The latter were characterized by a uniform arrangement of dislocations, which were less susceptible to annihilation during pre-annealing. The recovery of nickel oxide was accompanied by movement of the interparticle surface of the fusion, which led to the formation of a defective structure. In the process of NiO recovery, a complicated structure was formed in the volume of the migrating NiO-Ni boundary due to disoriented dislocations that did not disappear even at high sintering temperatures (Fig. 3).



a) *b)*

c) *d)*

Fig. 3. Microstructures of an alloy made of PZHRV 2.200.26 powder when Ni was introduced into the initial charge in the amount of: *a, b* — 20%; *c, d* — 40% at a sintering temperature of 1,150°C

The diffusion of nickel into powdered iron was most active along grain boundaries with a deviation deep into the grain. This was due to the lower activation energy of the grain boundary diffusion process compared to the bulk one. The high diffusion permeability of grain boundaries was due to their defective structure. The predominant dissolution of nickel along the boundaries of iron grains was more pronounced at a low sintering temperature (1,050°C), when volumetric diffusion was difficult (Fig. 4, 5).

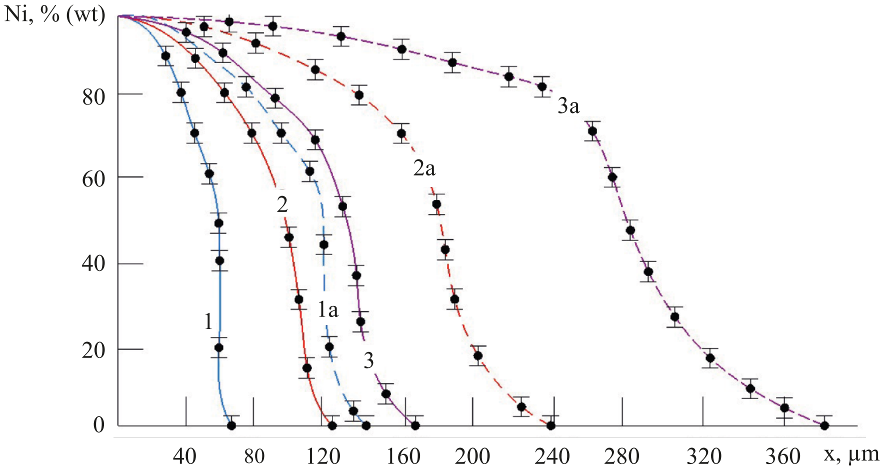
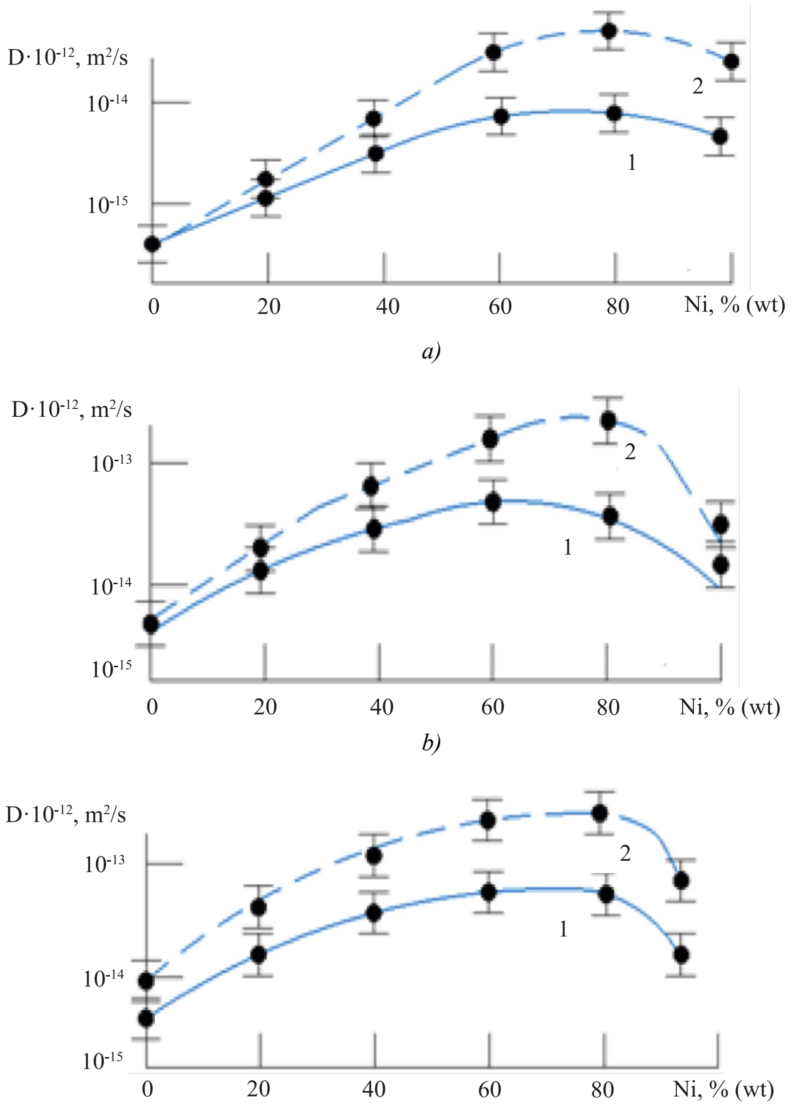


Fig. 4. Distribution of Ni concentration in the diffusion zone of Fe-Ni pairs (solid line 1, 2, 3) and Fe-NiO (dotted line 1 *a*, 2 *a*, 3 *a*) after sintering in a hydrogen atmosphere at temperatures:
1, 1 *a* — 1,050°C; 2, 2 *a* — 1,150°C; 3, 3 *a* — 1,250°C



a)

b)

c)

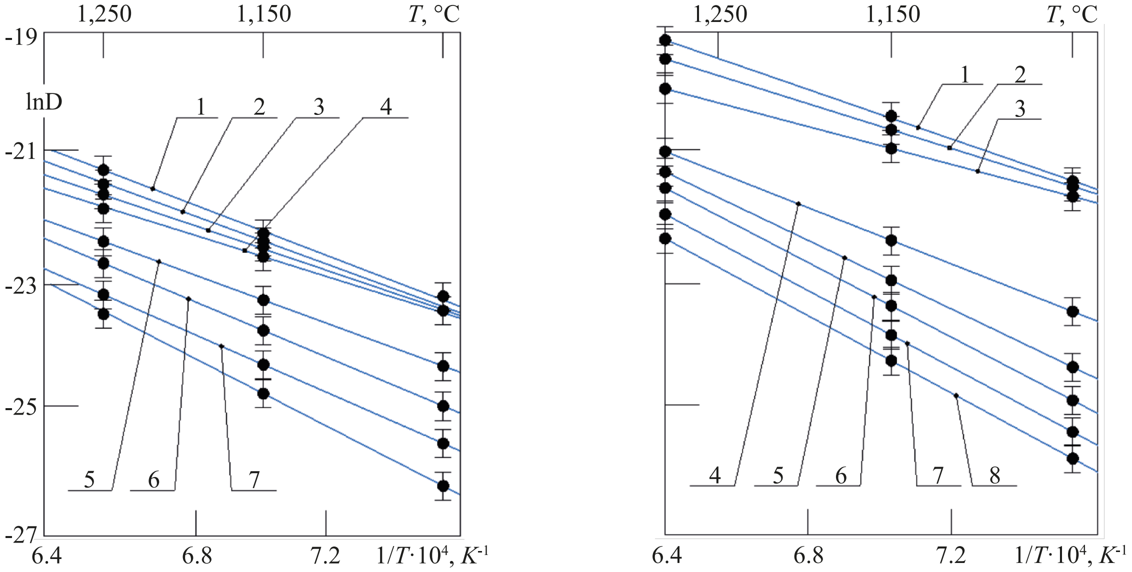
Fig. 5. Dependencies of the coefficients of mutual diffusion of Fe-Ni (line 1 and Fe-Ni (line 2) after annealing and sintering at temperatures:
a — 1,050°C; *b* — 1,150°C; *c* — 1,250°C

On all curves, the mutual diffusion coefficient had a maximum at a nickel concentration of more than 60%. These data coincided with the results of the work in which the coefficients of mutual diffusion were analyzed by finite element modeling of a simple model. At sintering temperatures of 1,050°C and 1,150°C in this concentration range, the mutual diffusion coefficient in the Fe-NiO pair was about 10 times higher than in the Fe-Ni pair [19]. With an increase in the sintering temperature to 1,250°C, this difference existed throughout the concentration range, which was also confirmed by the results of [18] and was shown in Figure 6.

Active diffusion energy $\overline{E}$ and pre-exponential multiplier $\overline{D_0}$ were determined based on the temperature dependencies of the mutual diffusion coefficients in accordance with the Arrhenius formula [6]:

$$\overline{D} = \overline{D_0}\,exp - \frac{\overline{E}}{RT} \qquad (1)$$

The diffusion energy in all the cases considered was found to be lower than the values reported in the literature for compact materials [19].



a)

b)

Fig. 6. Temperature dependencies of mutual diffusion coefficients:
a — Fe-Ni systems; *b* — Fe-Ni systems on NiO concentration:
1 — 90%; 2 — 80%; 3 — 70%; 4 — 50%;
5 — 40%; 6 — 30%; 7 — 20%; 8 — 10%

The porosity and structural activity of the nickel powder component influenced the increase in the processes of surface and grain boundary diffusion, which were realized by mechanisms with low activation energies, as well as diffusion by defects. The activation energy (Fig. 7) and the pre-exponential multiplier had a minimum in the concentration range of 50–70% Ni (Fig. 8). $\overline{E}$ values were suitable for Fe-NiO and NiO systems. Pre-exponential multiplier $\overline{D_0}$ in the Fe-NiO system was significantly greater than in the iron-nickel system, and increased the coefficient of mutual diffusion of elements during recovery annealing and sintering. Under thermomechanical action, as well as during the diffusion of carbon into the iron crystal lattice, the interparticle surface of the fusion was displaced, which could serve as the beginning of crack development [19]. This was due to an increased defect in the nickel structure after recovery, which was confirmed by X-ray analysis data [19]. The dislocation density of nickel powder recovered from oxide at a temperature of 700°C was 1.8·1,010. For carbonyl nickel it was $1.0\cdot10^8$ cm^{-2}.

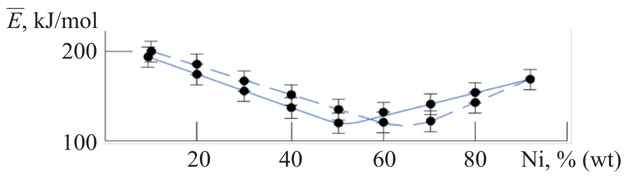


Fig. 7. Activation energy of mutual diffusion $\overline{E}$ in Fe-Ni systems (dotted line) and Fe-NiO systems (solid line)

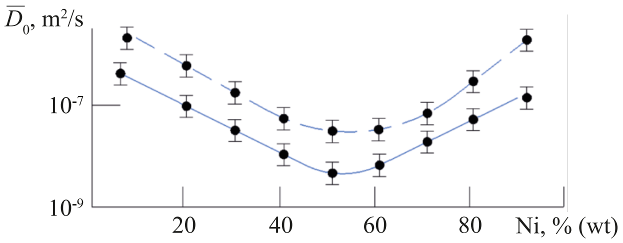


Fig. 8. Pre-exponential multiplier $\overline{D_0}$ (m^2/s) in Fe-Ni (dotted line) and Fe-Ni (solid line) systems

Discussion and Conclusion. The process of diffusion annealing of samples obtained from PZHRV 2.200.26 iron powder is accompanied by the so-called “Frenkel effect”, when atoms, ions or defects in a crystalline material can move or diffuse at elevated temperatures. During diffusion annealing, diffusion and recombination of defects such as vacancies (absence of an atom in the crystal lattice) and vacancy defects (atoms in the wrong places in the crystal lattice) are possible. As a result, the crystal can transform or get rid of these defects. When a vacancy and a vacancy defect are in close proximity, a Frenkel pair is formed. These pairs can move through the crystal via diffusion. If a Frenkel pair comes into contact with one of the vacancies, it can eliminate two defects and thereby purify the crystal of defects.

During the sintering process of alloyed powder steel, atoms diffuse between the powder particles, forming bonds at the interface. The diffusion of alloying elements in the powder steel leads to the formation of intermetallic compounds, as well as the creation of dispersed and basic phases. These diffusion processes can be accelerated by increasing the sintering temperature.

As a result of the studies performed, the quantitative parameters of Ni concentration distribution in the iron base were determined depending on the sintering temperature. The minimum distance over which nickel is distributed is 70 microns at a sintering temperature of 1,050°C. When sintered at 1,250°C, the distribution zone of pure nickel increases to 165 microns. However, when using nickel oxide, this zone increases above 360 microns at the same temperature. This indicates that the diffusion in a system with nickel powder recovered from oxide is 5–10 times higher than when carbonyl nickel powder is used. This may be important in the processes of alloying or saturating materials. The high rate of diffusion of recovered nickel can lead to faster and more uniform penetration of alloying elements into the material. This can be useful when creating a surface layer with certain properties or increasing the strength and other mechanical properties of the material.

The analysis of the results obtained indicates a different intensity of diffusion processes in powder alloyed steels. This is associated not only with the distortion of the crystal lattice of the starting materials, but also with an increased concentration of defects formed during the pressing of blanks. An understanding of diffusion processes and their influence on the formation of the structure of alloyed powder steels makes it possible to optimize sintering processes and obtain materials with desired properties and microstructure. Thus, the choice between nickel powder recovered from oxide and carbonyl nickel powder should be based on specific requirements and process conditions. Further research and experimentation can help to fully explore the potential of these options and determine the most effective approach for a given application.

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ZhV Eremeeva: preparation of samples for research on a scanning microscope, analysis of microstructure results and construction of mathematical dependencies.

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