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Original Empirical Research

Phase Transformations in Powder Sintered Steels during Cooling

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Abstract

Introduction. Heat treatment is a common post-processing operation applied to powder steel (PS) after deformation. The fundamental principles of heat treatment theory, developed for solid materials, also apply to PS. However, the specific structure of PS introduces quantitative and qualitative differences in the kinetics of heat treatment processes. Therefore, it is important to understand the effect of heat treatment on the structure and properties of PS when developing new materials. The aim of this study is to investigate phase transformations in sintered powder steels during cooling and to determine their mechanical properties after heat treatment.

Materials and Methods. The study used domestic powders of brands PZHRV 2.200.28 (TU 14-1-5365-98) and PL-N4D2M (TU 14-5402-2002) produced by PJSC Severstal (Cherepovets). During the mixing process, ultrafine additives of silicon nitride (Si3N4) and nickel oxide (NiO) manufactured by Plasnotherm (Moscow) were added to the charge. Before use, the powders were tested on a universal laser particle size measuring device (FRITSCH ANALYSETTE 22 MicroTec plus) and a submicron particle analyzer (Beckman COULTER No. 5). To prepare the charge, we used a two-cone mixer RT-NM05S (Taiwan) and an ultrasonic station for sieving and mixing powders with ultrafine particles Assonic SPC (China). Static cold pressing was carried out in laboratory molds on a hydraulic press model TS0500-6 (China) with a maximum force of 50 tons. Homogenizing sintering was performed in the laboratory of heat treatment at the Department of Materials Science and Technology of Metals at Don State Technical University in a muffle electric furnace model 6.7/1300 in the temperature range of 900–1150°C, in a protective gas environment — dissociated ammonia. Sintering time was 15–180 minutes. Heat treatment of sintered powder steels was also performed in these furnaces. Quenching of sintered samples was carried out at a temperature of 800°C. The initial porosity of sintered samples was 10.15.25%. Sintered samples were cooled at a temperature between 100 and 300°C. Tensile testing was conducted in accordance with GOST 18227–85[1], using a floor-mounted servohydraulic tensile testing machine MGS-V15 in an automatic mode, with the help of a personal computer. Hardness was measured using a Rockwell hardness tester TK-2M with a diamond cone indenter under a total load of 1471 N.

Results. The study conducted allowed us to identify the patterns of phase transformation in powder-sintered steels with ultra-fine particles during cooling after quenching. We experimentally determined the values of critical cooling points for powder-sintered eutectic steels at cooling rates of 60–400°C per minute. Additionally, we determined the mechanical properties of sintered powder steels with ultrafine particles depending on the temperature range of transformations

Discussion and Conclusion. The research has allowed us to establish the effect of ultrafine particles on the temperature of the critical points of sintered eutectoid PS, to construct diagrams of isothermal transformation of austenite, as well as to determine the mechanical properties of powder-sintered steels containing ultrafine particles. The analysis of the results obtained from the research has shown a multifaceted impact of nickel oxide and silicon nitride particles on phase transformations in powder-sintered steel.

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[1] GOST 18227-85 (ISO 2740-86). *Powder materials. Tensile test method.* URL: https://docs.cntd.ru/document/1200010599 (accessed: 12.04.2024). (In Russ.)

Keywords: sintered powder steels, ultrafine particles, critical cooling points, isothermal transformation, mechanical properties

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Оригинальное эмпирическое исследование

Фазовые превращения в порошковых спеченных сталях при охлаждении

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Аннотация

Введение. Термическая обработка является наиболее распространенной последеформационной операцией, применяемой к порошковым сталям (ПС). Принципиальные положения теории этого процесса, разработанные для компактных материалов, справедливы и для рассматриваемых ПС. Однако специфика структуры последних вносит количественные и качественные изменения в кинетику процессов, происходящих при различных видах термической обработки. Поэтому при разработке новых материалов необходимо уделять большое внимание влиянию термической обработки на их структуру и свойства. В связи с этим целью данного исследования является анализ фазовых превращений в порошковых спеченных сталях при охлаждении и определение их механических свойств.

Материалы и методы. В работе использованы отечественные порошки марок ПЖРВ 2.200.28 (ТУ 14-1-5365-98) и ПЛ-Н4Д2М (ТУ 14-5402-2002) производства ПАО «Северсталь» (г. Череповец). При смешивании в шихту добавлялись ультрадисперсные добавки нитрида кремния (Si_3N_4) и оксида никеля (NiO) производства компании «Плазнотерм» (г. Москва). Перед использованием порошки проходили контроль на универсальном лазерном приборе измерения размера частиц (модель FRITSCH ANALYSETTE 22 MicroTec plus) и анализаторе субмикронных частиц (Beckman COULTER № 5). Для приготовления шихты использовались двухконусный смеситель марки RT-NM05S (Тайвань) и ультразвуковая станция для просеивания и смешивания порошка с ультрадисперсными частицами Assonic SPC (Китай). Статическое холодное прессование проводилось в лабораторных пресс-формах на гидравлическом прессе модели TS0500-6 (Китай) с максимальным усилием в 50 тонн. Гомогенизирующее спекание проведено в лаборатории термической обработки кафедры «Материаловедение и технологии металлов» ДГТУ в муфельной электропечи модели SNOL 6,7/1300 в диапазоне температур 900–1150 °С в среде защитного газа — диссоциированного аммиака. Время спекания — 15–180 минут. В таких же печах производилась и термическая обработка спеченных порошковых сталей. Закалка осуществлялась на спеченных образцах при температуре 800 °С. Исходная пористость спеченных образцов составляла 10,15,25 %. Отпуск спеченных образцов проходил при температуре 100–300 °С. Испытания на растяжение проводились в соответствии с ГОСТ 18227–85 с использованием сервогидравлической напольной разрывной машины МГС-В 15 в автоматическом режиме с помощью персонального компьютера. Для измерения твердости использовался твердомер Роквелла ТК-2М с индентированным алмазным конусом при общей нагрузке 1471 Н.

Результаты исследования. В работе выполнены исследования, которые позволили определить закономерности фазовых превращений в порошковых спеченных сталях с ультрадисперсными частицами при охлаждении после операции закалки. Экспериментально определены значения критических точек охлаждения для спеченных порошковых сталей эвтектоидного состава для скоростей охлаждения 60–400 °С/мин. Определены также механические свойства спеченных порошковых сталей с ультрадисперсными частицами в зависимости от температурного интервала превращений.

Обсуждение и заключение. Исследования позволили установить влияние ультрадисперсных частиц на температуру критических точек спеченных ПС эвтектоидного состава, построить диаграммы изотермического превращения аустенита, а также определить механические свойства спеченных порошковых сталей с ультрадисперсными частицами. Анализ полученных результатов исследований показал разнонаправленное влияние частиц оксида никеля и нитрида кремния на фазовые превращения в порошковых спеченных сталях.

Ключевые слова: спеченные порошковые стали, ультрадисперсные частицы, критические точки охлаждения, изотермическое превращение, механические свойства

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Introduction. Heat treatment is understood as a set of heating, holding at high temperatures and cooling operations in order to change the structure and workability of the material, improve the combination of its mechanical and physical properties without changing the shape and size of the product. Heat treatment is an effective method of increasing physical and mechanical properties and wear resistance of steel [1].

Specific features of sintered steels (porosity, heterogeneity of structure, high oxidation capacity, etc.) make it difficult to use the technological modes of heat treatment developed for cast steels, although the main patterns of processes occurring during heating and cooling of compact steel can be transferred to sintered materials [2].

Studies of powder materials, including heat treatment, show a significant effect of porosity, as one of the features of their structure, on the development of phase transformations both during heating and cooling [3]. The presence of pores in powder materials and powder steels, as well as a high content of non-metallic inclusions, including those that modify the structure and density of dislocations, facilitate the formation of nuclei of new phases. This statement is supported by the results of thermodynamic analysis of phase transformations conducted on the basis of relevant data [4].

The aim of this work is to identify the features of the kinetics of phase transformations in powder sintered steels with ultrafine particles during cooling, to determine their mechanical properties depending on the temperature range. The main task of the study in this regard is to analyze the effect of porosity and cooling rate on the change in the position of critical points Ar_1 and Ar_3, as well as the effect of ultrafine additives on the initial phase of austenite decomposition and the mechanical properties of sintered powder steels

Materials and Methods. Powders of PZhRV 2.200.28[2] and N4D2M[3] brands by PJSC Severstal (Cherepovets) and ultrafine particles of nickel oxide and silicon nitride from Plasmotherm were used in the work[4]. Table 1 presents data on the total chemical composition.

Table 1

Chemical composition of powders

Powder grade	Mass content of components, %									
	Mo	Ni	C	O	H	Cu	Si	Mn	0P	S
PZhRV 2.200.26	–	–	0.090	0.140	–	–	0.014	0.087	0.012	0.005
N4D2M	0.400–0.500	3.600–4.400	0.020	0.025	–	1.300–1.700	0.050	0.150	0.020	0.020

Technological properties of PZhRV 2.200.26 powder: bulk density was from 2.4 to 3.0 g/cm^3, fluidity was no more than 37 sec/50 g, density at P = 700 MPa was 7.0–7.05 g/cm^3, strength at density of 6.5 g/cm^3 was more than 14 N/mm^2. The analog was the powders of the Swedish company Höganäs: AHC 100.29, NC 100.24, SC100.26, ASC100.29. Scope of application: shock absorption group parts, transmission parts, body parts, gears, connecting rods, couplings, bushings, etc.

According to TU 14-5402-2002, the powder of PL-N4D2M brand produced by Severstal contained 4% of nickel, 1.5% of copper and 0.5% of molybdenum. It was developed by specialists of Central Research Institute chermet named after Baikov. It was based on the iron powder of the PZHRV line. The final product was a partially alloyed mixture with excellent compaction (at a compacting pressure of 600 MPa, the density values were at least 7.1 g/cm^3).

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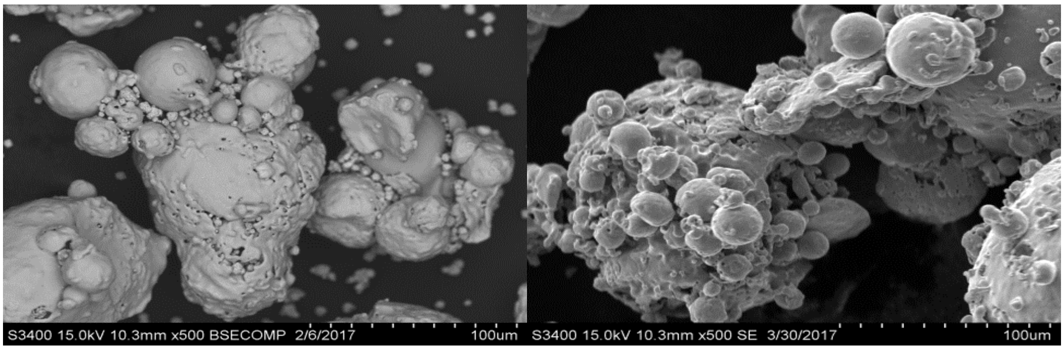
[2] TU 14-5365-98. *Iron powder, sprayed with air.* (In Russ.)
[3] TU 14-5402-2002. *Diffusion-doped iron powder.* (In Russ.)
[4] *Plasmotherm LLC company. Metal Nanopowders.* URL: https://plasmotherm.ru/catalog/metal/item_4.html (accessed: 12.04.2024). (In Russ.)

Figure 1 provides the morphology of the studied powders.



a) *b)*

Fig. 1. Powder particles: *a* — PL-N4D2M; *b* — PZhRV 2.200.26

Figure 2 shows a SEM image of ultrafine nickel oxide particles. Table 2 shows the properties of nickel oxide particles. The powder had a wide particle size distribution and represented individual spherical particles[5].

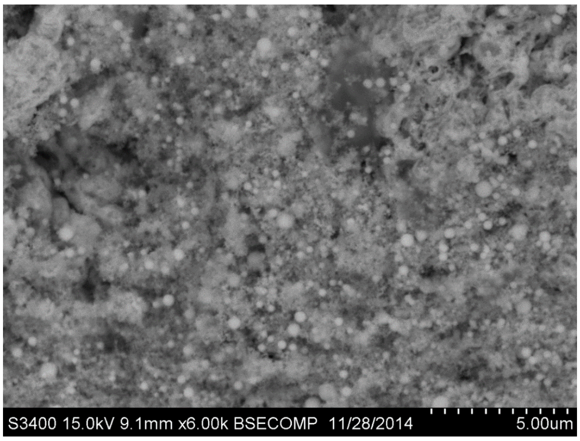


Fig. 2. SEM images of ultrafine NiO particles

Table 2

Main properties and characteristics of ultrafine nickel oxide powder

Production technology	Plasma chemical synthesis
Powder color	black
Particle size, nm	50–85
Average size of agglomerates, µm	30–80
Specific surface area, m^2/g	5–20
Content of the main component, %	99.8

Table 3 shows the characteristics of ultrafine nitride powder[6].

Table 3

Characteristics of ultrafine silicon nitride powder

Parameter	Value
Particle size, nm	10–30
Specific geometric surface, m^2/g	50–140
Content of the main component, %	99.75

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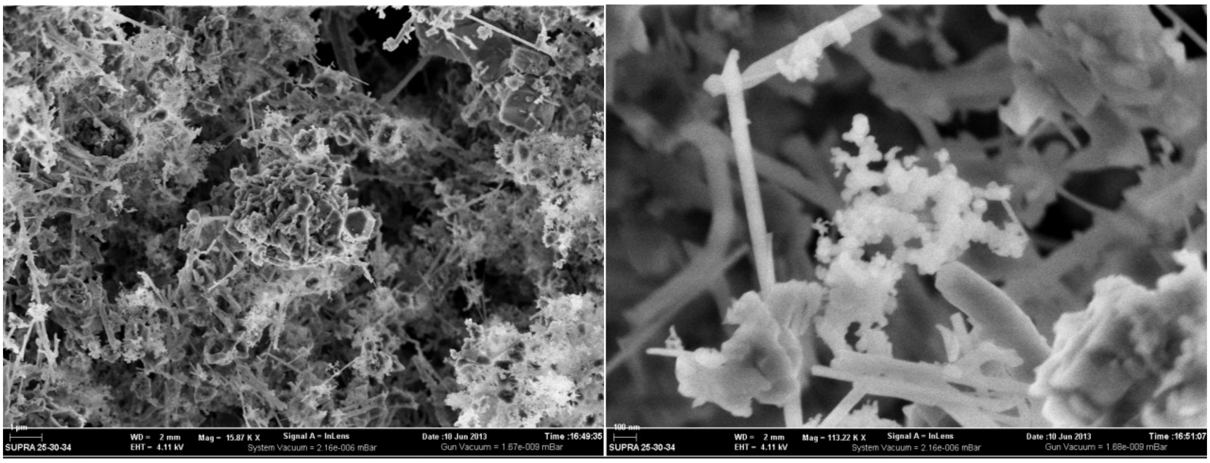
[5] TU 14-5402-2002 *Diffusion-doped iron powder.* (In Russ.)
[6] Id.

Ultrafine particles had a shape of needles (Fig. 3), formed fleecy flakes after contact with water and did not change size after ultrasonic treatment[7].



a) *b)*

Fig. 3. Image through a scanning electron microscope of ultrafine Si_3N_4 particles:
a — general view of the particle; *b* — particles at magnification 10^9

Experiments. Pressing was carried out using laboratory molds and a hydraulic press model TS0500-6 (China) with a maximum force of 30 tons. The prismatic blanks were molded in a mold shown in Figure 4. This mold consisted of container (1), two semi-matrixes (2), punch (3), inserts (4), which were made of tool steels U8, U10A. Their hardness after heat treatment reached 55–62 HRC. The residual porosity of the samples after pressing, depending on the applied pressure, was 10–40%. The prismatic blanks after molding had dimensions of 9.5x54.3 mm.

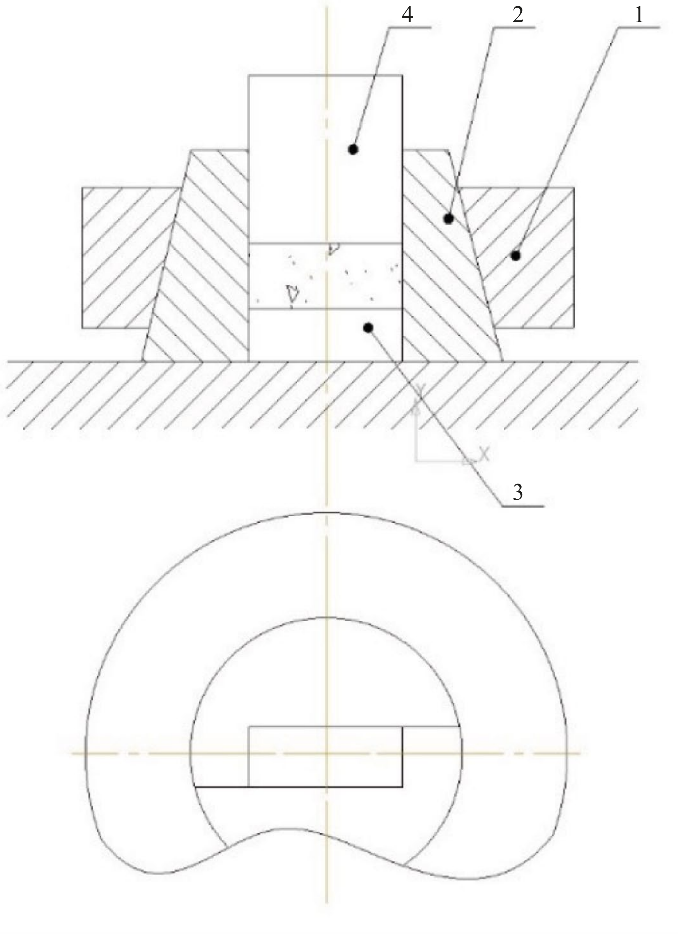


Fig. 4. Diagram of the mold for cold pressing of prismatic samples

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[7] TU 14-5402-2002 *Diffusion-doped iron powder.* (In Russ.)

Figure 5 shows a mold used for the production of tensile strength test samples.

Fig 5. Laboratory mold for the manufacture of tensile test samples according to GOST 8227-98
for cold-pressed and sintered samples

Sintering and heat treatment were carried out in the laboratory of heat treatment at the Department of Materials Science and Technology of Metals at Don State Technical University in a chamber electric furnace with protective atmosphere PKZ-1.0-7 (Russia) in a temperature range 900–1150 °C in a protective gas environment — dissociated ammonia (Fig. 6). Sintering time was 15–180 minutes.

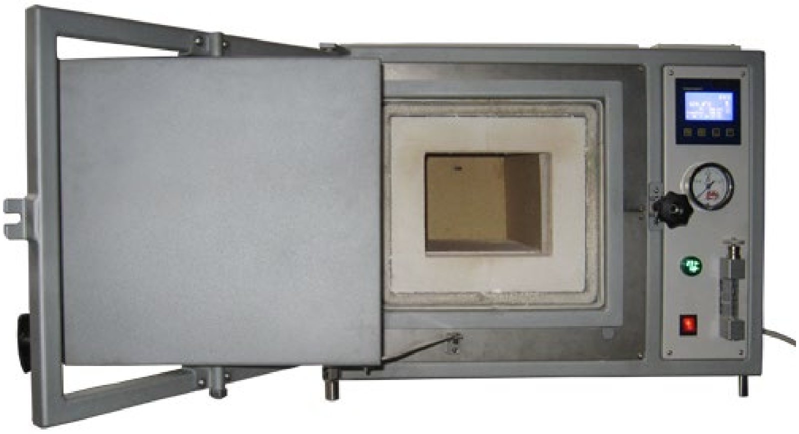
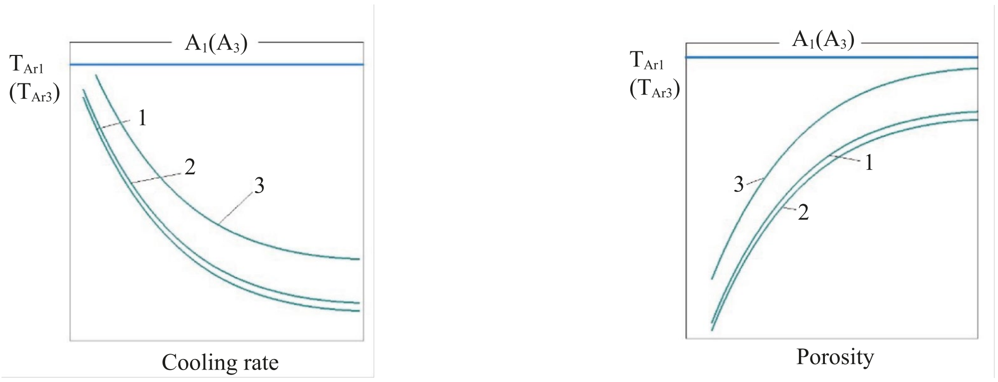


Fig. 6. Laboratory chamber electric furnace with protective atmosphere PKZ-1.0-7

Results. Let us imagine the process of phase transformations of powder sintered steels depending on their structural state. The dependence of the location of points A_{r1} and A_{r3} was established depending on such parameters as cooling rate ($V_{охл}$), porosity of samples and the introduction of ultrafine particles into the steel (Fig. 7). These parameters have different effects on the temperature ranges of phase transformations and the kinetics of austenite transformation [5]. A higher cooling rate slowed down, and greater porosity accelerated the transformation of austenite, shifting temperature ranges in opposite directions.

The effect of nickel oxide and silicon nitride additives was multidirectional [6]. If the first additive lowered the temperature of critical points, then the second one increased it. Table 4 provides the values of critical points for sintered PS of eutectoid composition for cooling rates of 60–400 °C/min. For PS based on N4D2M powder, the temperature of critical points was lower than that of iron powder, due to a slowdown in diffusion processes in the alloyed matrix [7].



a) *b)*

Fig. 7. Dependence of the position of points Ar_1 and Ar_3 for powder steels: 1 — clear; 2 — PS + 1 % NiO, 3 — PS + 0.2 % Si_3N_4; *a* — on cooling rate; *b* — on porosity

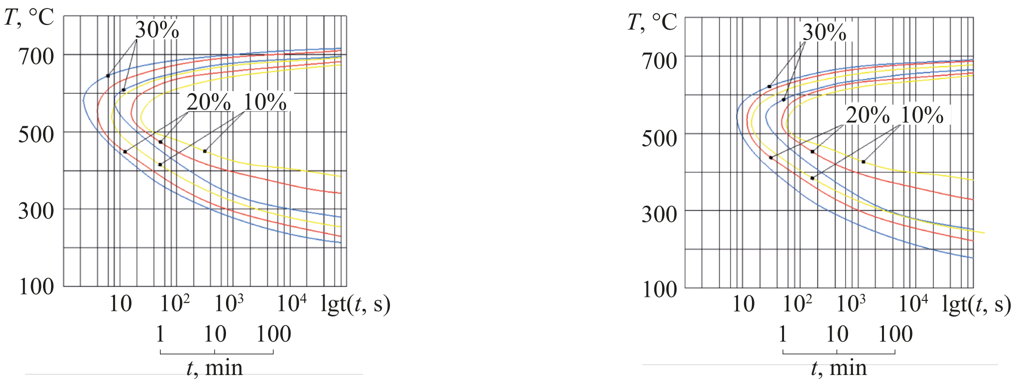
Table 4

Temperature points Ar_1 and Ar_3 of powder steels

Base of the alloy	Carbon content	Porosity, %	Point A_{r1}temperature, C	Point A_{r3}temperature
PZhRV 2.200.26	0.5	10	695*; 693**; 690***	725; 722; 719
		20	705; 702; 698	730; 727; 720
		30	712; 710; 708	737; 735; 732
	0.8	10	715; 713; 710	–
		20	718; 713; 708	–
		30	720; 716; 712	–
PZhRV 2.200.26 + 1% NiO	0.5	10	693; 690; 688	723; 720; 717
		20	703; 700; 696	728; 725; 718
		30	710; 708; 705	735; 732; 728
	0.8	10	712; 710; 708	–
		20	716; 711; 705	–
		30	723; 720; 716	–
PZhRV 2.200.26 + 0.2% Si_3N_4	0.5	10	700; 695; 693	730; 726; 722
		20	710; 707; 702	736; 732; 726
		30	718; 715; 712	740; 737; 735
	0.8	10	720; 722; 724	–
		20	723; 720; 717	–
		30	725; 722; 718	–
N4D2M	0.5	10	692; 690; 687	723; 719; 715
		20	700; 698; 695	726; 723; 716
		30	708; 705; 702	734; 730; 728
	0.8	10	710; 708; 702	–
		20	714; 711; 706	–
		30	717; 712; 708	–
N4D2M + 1% NiO	0.5	10	690; 686; 682	720; 717; 713
		20	700; 696; 692	725; 722; 716
		30	705; 703; 697	730; 728; 722
	0.8	10	707; 704; 698	–
		20	711; 708; 706	–
		30	715; 710; 706	–
N4D2M + 0.2% Si_3N_4	0.5	10	696; 692; 688	724; 720; 718
		20	706; 702; 698	732; 728; 722
		30	714; 710; 706	736; 730; 725
	0.8	10	716; 714; 710	–
		20	718; 720; 722	–
		30	720; 722; 726	–
*Note: cooling rates, °/min: *–60; **–200; ***–400*				

Upon slow cooling, austenite decomposed into a eutectoid ferrite-cementite mixture, the dispersion of which depended on the degree of supercooling [8]. During the incubation period, supercooled austenite remained stable, but after that, its decay began. C-shaped curves were used to describe the decay of austenite (Fig. 8) [9].

The analysis of the diagrams of isothermal transformation of austenite of powdered steels PZhRV 2.200.26 + 0.8%C and N4D2M + 0.8%C showed that with increasing porosity, the stability of supercooled austenite decreased, as evidenced by the displacement of C-shaped curves to the left. The area of minimum stability of austenite moved towards elevated temperatures. The total time of isothermal decomposition of austenite increased.



a) *b)*

Fig. 8. Diagram of isothermal transformation of austenite samples with porosity of 10, 20, 30%: *a* — from steel PZhRV 2.200.26 + 0.8% C; *b* — from steel N4D2M + 0.8 %

When using an alloyed powder, the C-shaped curves shifted to the left and to the region of lower temperatures, which confirmed the theoretical position about the diffusive nature of the formation of a ferrite-cementite mixture during the decomposition of austenite. How did ultrafine additives (0.2 % Si_3N_4 and 1 % NiO) affect the decomposition of austenite? To determine this, experiments were conducted in the field of minimal austenite stability. The results are shown in Figure 9. The solid line is steel based on PZhRV 2.200.26 powder, the dashed line is steel based on N4D2M powder.

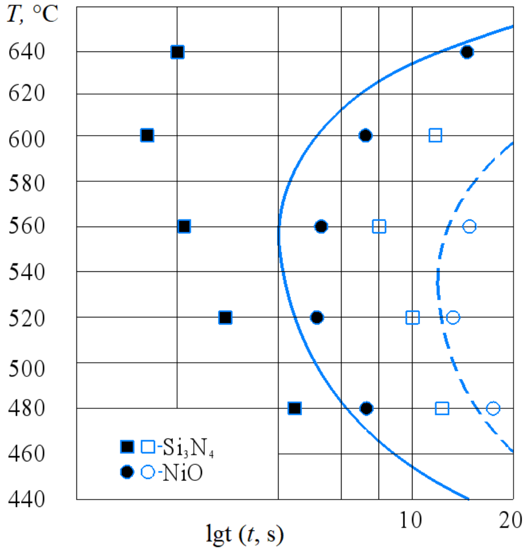


Fig. 9. The effect of ultrafine additives in steel based on PZhRV 2.200.26 powder (dashed markers) and in steel based on N4D2M powder (unshaded markers) on the initial phase of austenite decomposition

The addition of silicon nitride promoted the decomposition of austenite, increasing the temperature of the minimum stability of austenite, and shortening the incubation period [9]. This was due to the creation of areas of facilitated appearance of ferrite and iron carbide nuclei by the inclusions of the additive. The addition of nickel oxide led to an increase in the nickel content in the solid solution, which slowed down the diffusion processes and the decomposition of austenite.

It was shown in [10] that in the temperature range 700-600 °C austenite decomposed into lamellar perlite. At a temperature of minimal stability, the product of austenite decomposition was troostite, and in the temperature range from 500 to 250 °C, austenite transformation occurred with the formation of bainite.

In proeutectoid steels, the decomposition of austenite was preceded by the release of ferrite. At the same time, the pore surface was an area of predominant release and growth of ferritic inclusions, compared with high-angle boundaries [11]. Such a mechanism of nucleation of ferrite grains caused the absence of a Widmanstatten structure that reduced mechanical properties. The release of the excess ferritic component occurred in stages. First, a micro-volume with a crystalline structure close to the equilibrium structure of ferrite was formed. Then this micro-volume was transformed into a stable center of a new phase by diffusive changes in the chemical composition [12].

The peculiarity of the powder structure of steel, namely the presence of free pore surfaces with increased diffusion mobility of atoms, led to the predominant nucleation of the ferritic phase on these surfaces, which was reflected in the displacement of C-curves of austenite transformation in the diagrams. Table 5 shows the mechanical properties of sintered PS (porosity 10%) depending on the temperature range of transformation [13].

Table 5

Mechanical properties of sintered powder steels

Steel composition	Temperature range, °C	Mechanical properties		
		$\sigma_{в}$, MPa	HRB	δ, %
PZhRV 2.200.26 + 0.8% °C	700–680	220	70	11
	650–630	260	82	13
	600–550	280	88	13
PZhRV 2.200.26 + 0.8% °C + 1% NiO	700–680	222	71	11
	650–630	262	83	12
	600–550	281	89	12
PZhRV 2.200.26 + 0.8% °C + 0.2% Si_3N_4	700–680	270	85	10
	650–630	290	90	10
	600–550	310	95	9
N4D2M + 0.8% °C	680–660	780	80	8
	630–600	795	83	9
	570–540	800	87	10
N4D2M + 0.8% °C + 1% NiO	680–660	850	92	11
	630–600	860	103	11
	570–540	880	106	12
N4D3M + 0.8% °C + 0.2% Si_3N_4	680–660	810	102	7
	630–600	860	105	8
	570–540	880	108	8

Discussion and Conclusion. As a result of the studies, it was found that increasing the cooling rate reduced the temperature range of austenite decomposition. This, in turn, led to an increase in the discreteness of the ferrite-cementite mixture, which resulted in an increase in tensile strength and hardness [14]. The addition of nickel oxide to the charge based on PZhRV 2.200.26 powder had a negligible effect on the mechanical properties of steel. The beneficial effect of this additive was manifested at the sintering stage, during which the oxide was reduced and nickel dissolved in the iron matrix. Cooling after sintering led to the formation of a pearlitic structure without metallographically detectable dispersed hardening inclusions. Therefore, during subsequent heat treatment based on austenite-pearlite transformation, there was no hardening effect from additional inclusions. When using N4D2M powder, the effect of the nickel oxide additive introduced into the charge was more noticeable. This circumstance was explained by the concentration of nickel in the ferritic component up to 5% and an increase in the efficiency of ferrite hardening with an increase in the nickel content in solid solution [15]. Unlike nickel oxide, silicon nitride particles that were added to the charge remained as inclusions in the steel's structure, contributing to the formation of new phase nucleation during austenite decomposition. This contributed to increased tensile strength and hardness through the effect of dispersion hardening [16]. The analysis of diagrams of isothermal transformations of austenite of powdered steels PZhRV 2.200.26 + 0.8%C and N4D2M + 0.8% with a porosity of 10, 20, 30% was carried out. When using an alloyed powder, the C-shaped curves shifted to the left and to the region of lower temperatures, which confirmed the theoretical position about the diffusive nature of the formation of a ferrite-cementite mixture during the decomposition of austenite. The dependencies of the mechanical properties of sintered powder steels with ultrafine particles on the temperature range of transformations were determined.

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