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Influence of Carbon Content on the Formation of a Contact Interparticle Surface during Hot Post-Pressing

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Abstract

Introduction. The technology for producing hot-formed powder steel is one of the most energy-intensive in powder metallurgy, which includes a large number of operations. The study of the influence of technological modes on the final properties of the part is an urgent task. Developed by the scientific team under the leadership of Yu.G. Dorofeev at the end of the XX century, the technology of manufacturing hot-formed powder steels is currently one of the main ones in the production of high-density products. However, the use of new materials that improve the mechanical properties of products requires a modern approach to analyzing the quality of interparticle splicing of powder particles. The influence of the following technological factors on the formation of qualitative interparticle splicing was established: the blank density, the granulometric composition of the initial charge, the temperature and holding time of the blank during heating, the ratio of its dimensions, the deformation rate. The study objective is to analyze the effect of a graphite-containing component on the mechanical properties of hot-formed powder alloys due to the formation of high-quality interparticle splicing.

Materials and Methods. The work used domestic and foreign powders produced by PAO Severstal and the Swedish company Höganäs with the addition of carbon GC-1 (GOST 4404-78). Hot stamping was carried out on a crank press of the K2232 model with a maximum force of 1600 kN. The heating temperature of the workpieces varied between 800–1200 °C.

Results. As a result of the experiments, the influence of the sintering duration on the mechanical properties of materials was established. The reason for the change in mechanical properties are local inclusions of graphite, which did not have time to homogenize as a result of prolonged sintering. Technological modes of hot stamping for steels have been developed that affect the preservation or destruction of the pre-formed contact interparticle surface.

Discussion and Conclusion. The studies have shown that additional hot plastic deformation contributes to the formation of intracrystalline fusion on the entire contact surface. The addition of graphite to the charge improves splicing for alloyed iron powder and practically does not affect the use of alloyed and unalloyed iron powder.

Keywords: hot stamping, splicing, plastic deformation, surface microstructure

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Влияние содержания углерода на формирование контактной межчастичной поверхности при горячей допрессовке

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Аннотация

Введение. Технология получения горячедеформированной порошковой стали является одной из самых энергозатратных в порошковой металлургии, которая включает в себя большое количество операций. Изучение влияния технологических режимов на конечные свойства детали является актуальной задачей. Разработанная научным коллективом под руководством Ю.Г. Дорофеева в конце XX века технология изготовления горячедеформированных порошковых сталей на сегодняшний день является одной из главных в производстве высокоплотных изделий. Однако применение новых материалов, улучшающих механические свойства изделий, требует современного подхода к анализу качества межчастичного сращивания порошковых частиц. Установлено влияние на процесс формирования качественного межчастичного сращивания следующих технологических факторов: плотности заготовки, гранулометрического состава исходной шихты, температуры и времени выдержки заготовки при нагреве, соотношения ее размеров, скорости деформации. Целью данного исследования является анализ влияния графитсодержащего компонента на механические свойства горячедеформированных порошковых сплавов за счет формирования качественного межчастичного сращивания.

Материалы и методы. В работе использовались отечественные и зарубежные порошки производства ПАО «Северсталь» и шведской фирмы Högans с добавлением углерода ГК-1 (ГОСТ 4404-78). Горячая штамповка осуществлялась на кривошипном прессе модели K2232 с максимальным усилием 1600 кН. Температура нагрева заготовок варьировалась в пределах 800–1200 °С.

Результаты исследования. В результате проведенных экспериментов было установлено влияние продолжительности спекания на механические свойства материалов. Причиной изменения механических свойств являются локальные включения графита, которые не успели гомогенизироваться в результате длительного спекания. Разработаны технологические режимы горячей штамповки для сталей, влияющие на сохранение или разрушение предварительно сформированной контактной межчастичной поверхности.

Обсуждение и заключения. Исследования показали, что дополнительная горячая пластическая деформация способствует формированию внутрикристаллитного сращивания на всей контактной поверхности. Добавление в шихту графита способствует улучшению сращивания для легированного железного порошка и практически не сказывается при использовании легированного и нелегированного железного порошка.

Ключевые слова: горячая штамповка, сращивание, пластическая деформация, микроструктура поверхности

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Introduction. A formation feature of powdered porous blanks is the structure formation during their processing, which has a fundamental difference from the corresponding processing of monolith materials. The features of the technological processes of structure formation in powder metallurgy are due to the intense thermomechanical effect on the processed powder steel. A large number of processes of forming and structuring of material under conditions of

short-term thermomechanical action leads to the synthesis of scientific approaches of hot deformation in powder metallurgy, materials science, pressure welding, metal processing under pressure, theory of strength, plasticity [1–3].

The main objective of the authors of the article is to study the technological modes of hot deformation of products with different carbon content, as well as to study the microstructure in various zones of deformable products.

Materials and Methods. The formation of the contact surface of the powder material at the hot stamping stage is considered from the position of its initial state, which changes during the compaction process, i.e. the sequential increment of the contact surface.

Iron powders produced by the Höganäs (Sweden) and PAO Severstal (Russia) were used in the work [2, 4] (Table 1).

Table 1

Types and characteristics of the powders used

Powder brand	Manufacturer country	Powder production method
PZhRV2.200.26 TU 14-1-5365-98	RF, PAO Severstal	Atomization of the alloy by a stream of compressed air, recovery annealing
PL-N4D2M TU 14 -5402 2002	RF, PAO Severstal	Diffusion-reduction annealing of atomized powder
ABC100.30	Sweden, Höganäs	Spraying of iron melt
Astaloy 85Mo	Sweden, Höganäs	Spraying with water of an alloyed melt containing 0,85 % Mo
Distaloy HP-1	Sweden, Höganäs	Double diffusion alloying of powder Astaloy 85Mo:1.5 % Mo+4 % Ni,2 % Cu

Data on the total chemical composition are presented in Table 2.

Table 2

Chemical composition of the powders under study

Powder brand	Content of elements, mass content, %								
	C	O	Mo	Ni	Cu	Mn	Si	S	P
PZhRV2.200.26	0.02	0.25				0.15	0.05	0.015	0.02
PL-N4D2M	0.02	0.25	0.4-0.5	3.6-4.4	1.3-1.7	0.15	0.05	0.02	0.02
ABC100.30	0.001	0.04	–	–	–	0.06	0.007	0.01	0.004
Astaloy 85Mo	<0.01	0.07	0.85	–	–	0.06	0.008	0.02	0.005

The main alloying element in the studied material is carbon introduced into the charge in the form of graphite pencil GK-1 (GOST 4404-78). Table 3 provides the chemical composition.

Table 3

Chemical and granulometric composition of graphite powder

Name of indicators	Powder
	GK-1
Moisture content, mass., %	2.0
Ash content, mass., %	5.0
Sulfur content, mass., %	1.0
Granulometric composition, microns	+100-300

The formation of the interparticle splicing surface is influenced by inclusions of the second phase, in particular, graphite particles not dissolved in austenite [2, 4, 5]. Based on the results of the chemical analysis of the material, an understanding of the process of carbon dissolution in the sample is formed, which allows us to assume that complete dissolution of carbon takes no more than 60 minutes. The mechanical properties data presented in Table 4 indicate that for samples containing 0.5% C (mass fraction) this time is insufficient, since it does not provide a high level.

Table 4

Dependence of the mechanical properties of hot-deformable alloys on the duration of sintering

Metal base of the charge	Duration of sintering at 1000° C, hours	Mechanical properties after postcompaction		
		σ_B , MPA	Ψ , %	KCU, MJ/m ²
Astaloy 85Mo+0.5% C	0.5	610	0	0
	1	640	0	0
	1.5	690	35	0.38
ABC100.30+0.5%C	0.5	350	0	0
	1	370	0	0
	1.5	450	45	0.7

The data presented in Table 4 show that the following indicators are most sensitive to the degree of homogenization of alloys: ductility of the material and impact resistance.

The formation of sufficiently high mechanical properties of the materials under consideration with the addition of carbon occurs when choosing the correct sintering modes. The complete homogenization of carbon in the iron matrix of the base depends on this. To explain this dependence, the study of blank fractures by Auger-electron spectroscopy was carried out on a PHJ-680 spectrometer by Physical Electronics [2, 5, 6]. Figure 1 shows a fractogram of the surface of a sample sintered for 60 minutes. The area of this surface, indicated by point 8, deserves attention.

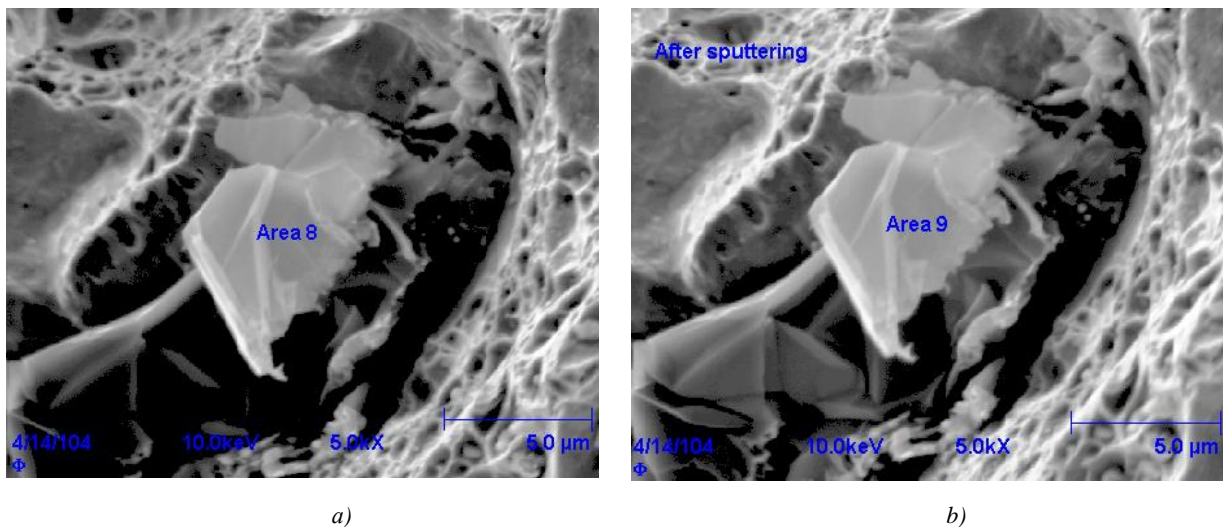


Fig. 1. Inclusion on the destruction surface of the blank.
Sintering at 1000 °C for 1 hour: *a* — before argon etching; *b* — after argon etching

The results of argon etching of the sample at point 8 shows that this morphological structural element is a region with a nonequilibrium carbon content (Fig. 2).

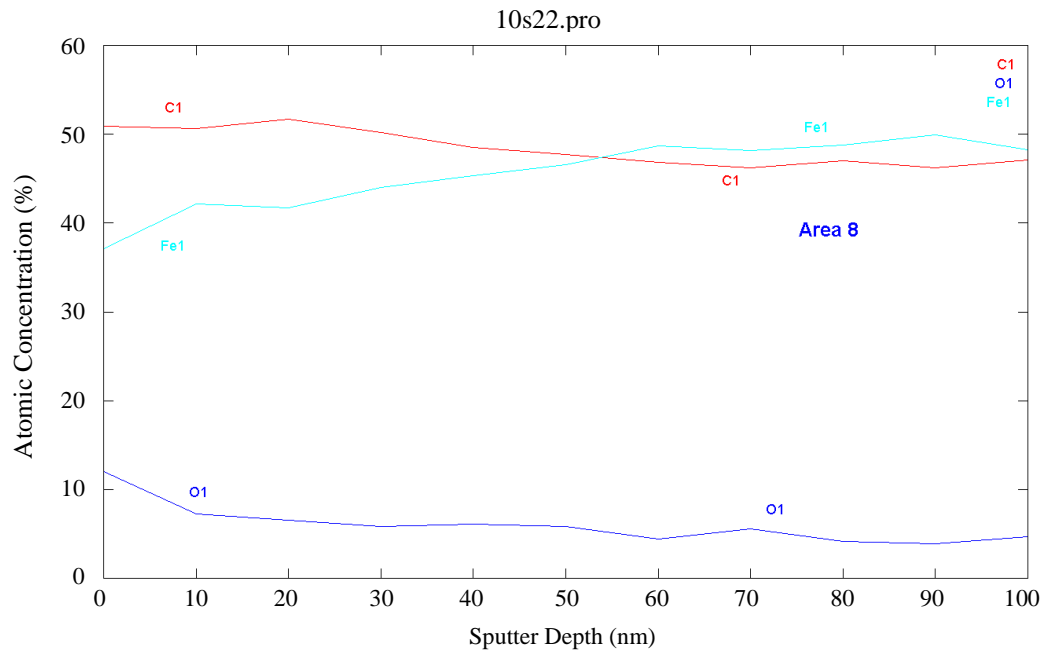


Fig. 2. Dependence of the distribution of elements at point 8 on the etching depth

The carbon content is about ~40 at % when etched into the depth of the surface of 100 nm. Consequently, the area under consideration is characterized by a three-dimensional inclusion formed as a result of diffusion during sintering of iron ions into a former graphite particle. Judging by the results of Auger-electron spectroscopy, its chemical composition corresponds to the formula of nonequilibrium carbide $\text{Fe}_{0.9}\text{C}$. A more even fracture surface of the considered zone indicates that the destruction of the sample occurred by the mechanism of chipping, characteristic of brittle fracture [1, 7, 8]. The structure, in which such an element is located, entails reduced mechanical properties of the alloys in question

Results. The research of the dependence of the mechanical properties of hot-formed steels on the carbon content in the charge were carried out. Pre-samples were sintered at a temperature of 1000 °C for 1.5 hours. Further, there was post-pressing to porosity values close to zero at a temperature of 1050 °C. This sintering mode ensures complete dissolution of carbon in the iron base of steel. The values of the ultimate strength, elongation and Vickers hardness of hot-formed steels with different carbon content are shown in Fig. 3.

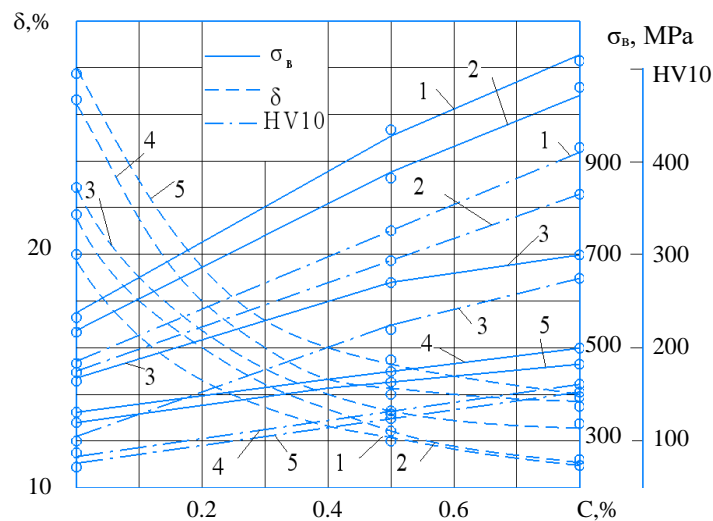


Fig. 3. Influence of carbon content on the mechanical properties of hot-formed steels based on powders:
1 — Distaloy HP-1; 2 — N4D2M; 3 — Astaloy 85Mo; 4 — PZhr2.200.26; 5 — ABC100.30

The nature of the presented dependencies is consistent with the theoretical provisions of classical and powder materials science [2, 9, 6], which is confirmed by the results of microstructural analysis (Fig. 4). With an increase in the carbon content in the steel composition, the amount of the ferritic component decreases and the amount of ferrite-cementite eutectoid mixture increases. With a carbon content of 0.8% C, the steel structure consists of finely dispersed troostite.

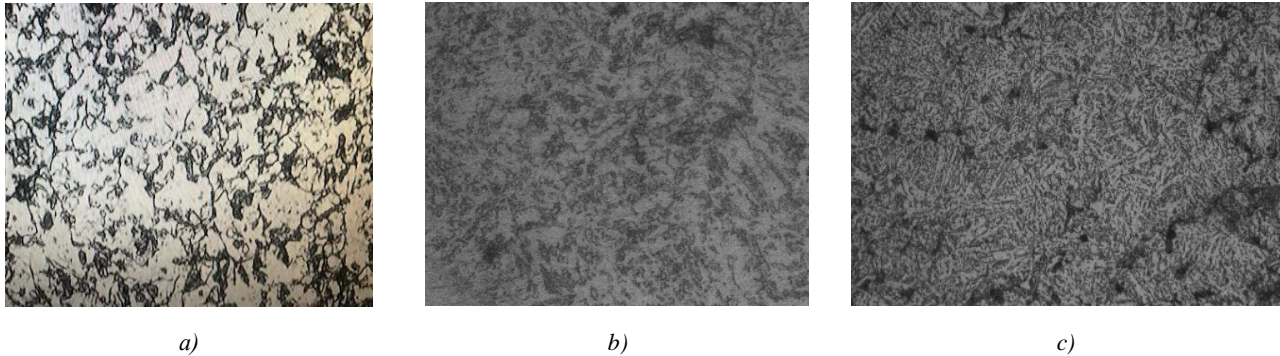


Fig. 4. Microstructure of hot-deformed powder steel based on PZhRV2.200.26 powder with different carbon content, x250:
a — 0.2 % C; b — 0.5 % C; c — 0.8 % C

There are no pores on the microsections, this fact indicates an almost non-porous state of the material.

The influence of carbon on the development of the contact surface is considered on the dependence of the mechanical properties of powder steel on the initial porosity (Fig. 5). Tested steels with a carbon content of 0.5% (wt.) were obtained by hot post-pressing at 1050 °C.

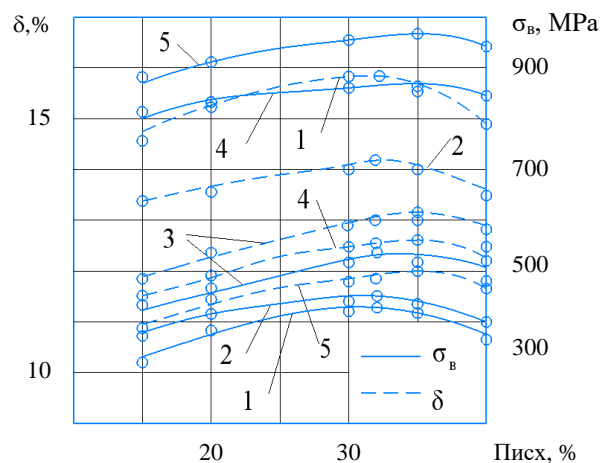


Fig. 5. Dependence of the mechanical properties of hot-deformed steels on the initial porosity:
1 — ABC100.30; 2 — PZhR2.200.26; 3 — Astaloy 85Mo; 4 — N4D2M; 5 — Distaloy HP-1

Comparing the data presented in Fig. 4 and Fig. 5, we can note the identical nature of the dependence of both strength and plastic properties on the initial porosity. For steels based on powders PZhR2.200.26 and AVS100.30, the extremum of properties is observed at the initial porosity of the workpiece of 30 %. In steels based on Astaloy 85Mo, N4D2M and Distaloy HP-1 powders, the extremum of properties shifts towards an increase in the value of the initial porosity. This circumstance can be interpreted as an increase in the quality of interparticle interaction, which is reflected in the position of the line delimiting the areas of technological modes characterized by partial or complete destruction or preservation and development of the pre-formed contact surface at the stage of hot pressing (Fig. 6) [10–12].

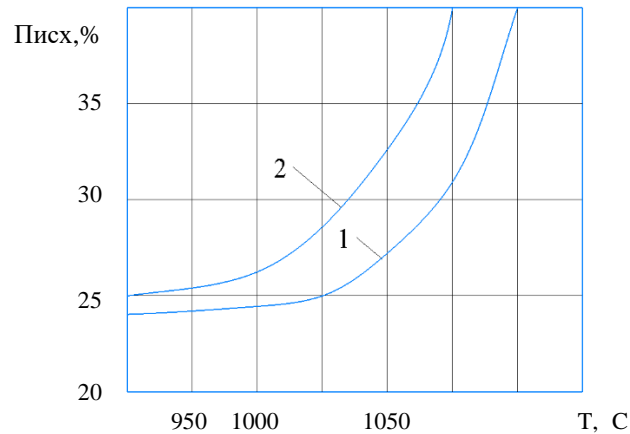


Fig. 6. Areas of technological modes of hot stamping affecting the preservation or destruction of the pre-formed contact interparticle surface for steels: 1 — PZhR2.200.26+0.5 % C; ABC100.30+0.5 % C; 2 — Astaloy 85Mo+0.5 % C; N4D2M+0.5 % C; Distaloy HP-1+0.5 % C

Table 5 provides a comparison of the results presented in Figures 5 and 6.

Table 5

Determination of the areas of technological modes of hot post-pressing for powder steels

Initial powder	Carbon content, %	Marking of the separation line
PZhR2.200.26; ABC100.30; Astaloy 85Mo; N4D2M; Distaloy HP-1	0	Line 1
PZhR2.200.26+0.5 % C; ABC100.30+0.5 % C; 2 — Astaloy 85Mo+0.5 % C; N4D2M +0.5 % C; Distaloy HP-1+0.5 % C	0.5	Line 1
Astaloy 85Mo; N4D2M; Distaloy HP-1	0.5	Line 2

The addition of carbon powder to the charge practically does not affect the application of optimal hot post-pressing modes for materials based on unalloyed iron powders. In the case of alloying iron powders with molybdenum, copper, nickel, when graphite is introduced into the charge and pre-sintering until complete homogenization of austenite, the recommended value of the initial porosity is shifted towards higher values.

It is possible to evaluate the quality of interparticle splicing of hot-deformed steels by the value of the Young's modulus. The value of the Young's modulus is taken as the criterion for additionally forged samples of the studied steels to a nonporous state (Table 6) [2, 5, 13].

Table 6

Parameters of powder steels in a nonporous state

Material		Density in the nonporous state, g/cm ³	Young's module, GPa
Metal base	Carbon content, %		
PZhRV2.200.26	0.5	7.79	201
ABC100.30		7.79	201
Astaloy 85Mo		7.83	206
N4D2M		7.81	203
Distaloy HP-1		7.85	208

Let us consider the dependence of the parameters of hot-formed steels with a carbon content of 0.5 % on the temperature of the hot post-pressing modes and the specific compaction work (W). As the parameters of the steels, we use the values of density (ρ), the Young's modulus (E) and the relative contact surface with intracrystalline splicing (α_{BKC}) (Table 7).

Table 7

Parameters of hot-deformed steels from hot post-pressing modes

Material	T, °C	W, MJ/m ³	ρ , g/cm ³	E, GPa	α_{BKC}
PZhRV2.200.26	950	60	6.5	112	0.56
		100	7.3	189	0.94
		120	7.5	193	0.96
		160	7.75	194	0.965
		180	7.79	195	0.97
	1050	60	7.25	181	0.9
		80	7.6	193	0.96
		100	7.75	195	0.97
		120	7.79	196	0.975
	1150	60	7.5	190	0.945
		80	7.74	196	0.975
		100	7.79	197	0.98
ABC100.30	950	60	6.6	166	0.82
		100	7.35	184	0.92
		120	7.55	189	0.94
		160	7.79	195	0.97
	1050	60	7.4	187	0.93
		80	7.68	194	0.965
		100	7.79	197	0.98
	1150	60	7.63	194	0.965
		80	7.79	198	0.985
Astaloy 85Mo	950	60	6.57	168	0.81
		100	7.49	191.2	0.92
		120	7.57	193.4	0.94
		160	7.81	199	0.96
		170	7.83	200	0.97
	1050	60	7.42	190.5	0.92
		80	7.69	197.5	0.96
		100	7.83	201	0.975
	1150	60	7.65	197	0.96
		80	7.83	202	0.98
N4D2M	950	60	6.53	165	0.81
		100	7.32	187	0.92
		120	7.52	190	0.93
		160	7.77	197	0.97
		180	7.81	198	0.98
	1050	60	7.28	185	0.91
		80	7.62	194	0.95
		100	7.77	198	0.97
		120	7.81	199	0.98
	1150	60	7.53	193	0.95
		80	7.76	199	0.98
		100	7.81	200	0.985
Distaloy HP-1	950	60	6.67	172	0.82
		100	7.4	190	0.91
		120	7.59	195	0.94
		160	7.85	202	0.97
	1050	60	7.47	193	0.93
		80	7.73	200	0.96
		100	7.85	203	0.975
	1150	60	7.69	200	0.96
		80	7.85	204	0.98

The experimental results presented in Table 7 show that in the entire temperature range of the study, a value of the density of the material corresponding to its nonporous state is achieved (Table 8).

Table 8

Specific compaction work (MJ/m^3) to achieve a nonporous state

T, °C	Iron base of powder steel with a content of 0.5 % C				
	PZhRV2.200.26	ABC100.30	Astaloy 85Mo	N4D2M	Distaloy HP-1
950	180	160	170	180	160
1050	120	100	100	120	100
1150	100	80	80	100	80

Discussion and Conclusion. Despite the achievement of a nonporous state, the values of the Young's modulus show that there are the opportunities to improve the functional properties of materials, that is, during hot post-pressing, the formation of intracrystalline splicing on the entire contact surface is not achieved. Figure 7 shows a fractogram of a destroyed sample of hot-deformed steel based on PZhRV2.200.26 powder with a density of 7.81 g/cm^3 . The fracture is characteristic of the viscous fracture of steel. Ridges and depressions of the pit relief are visible, which are the result of intense plastic deformation in the crack propagation center. With the predominance of the pit relief on the fractogram, areas with a flat relief characteristic of intercrystalline or transcrystalline fracture are observed. The presence of such zones on the fracture of steel indicates the incompleteness of interparticle splicing. The areas of intercrystalline cleavage directly indicate the absence of transformation of the interparticle splice surface into a large-angle intergranular surface. In the case of identification of flat fracture zones as a consequence of crack development by the mechanism of transcrystalline fracture, one can assume the hereditary nature of the structure in the fracture zone.

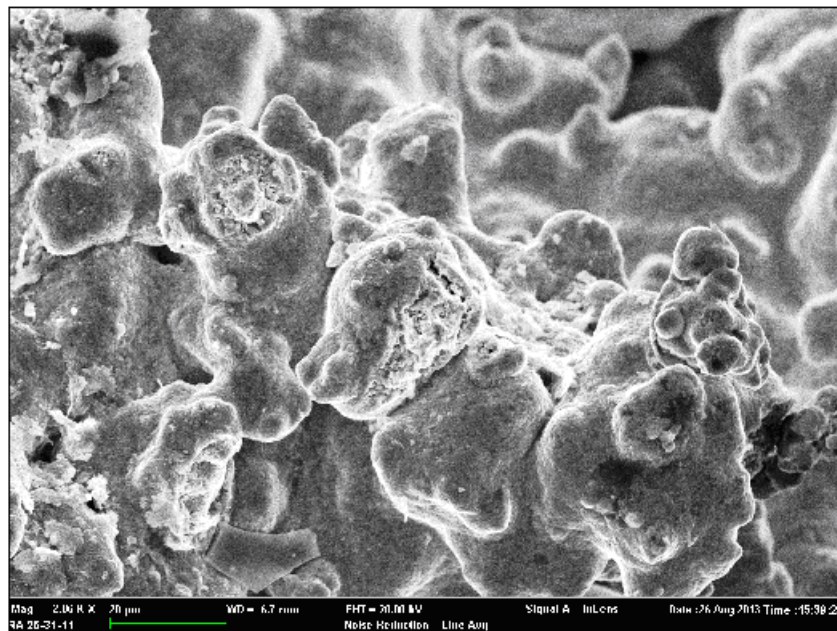


Fig. 7. Fracture surface of powder steel

The separation of the interparticle splice surface from submicropores, segregation atmosphere, dispersed inclusions of another phase leads to the preservation of these morphological elements of the structure in the zone of the former interparticle contact surface, which are factors contributing to the nucleation of the crack nucleus and its spread.

In relation to the studied steels with a content of 0.5% C, we use the technological technique proposed in [14, 15], which consists in additional hot plastic deformation with the determination of the critical degree of deformation. Let us consider the results of additional plastic deformation of a powder material based on powders PZhRV2.200.26 and N4D2M, carried out at a temperature of 1050 °C (Fig. 8). The Young's modulus is used as a criterion for the formation of intracrystalline fusion.

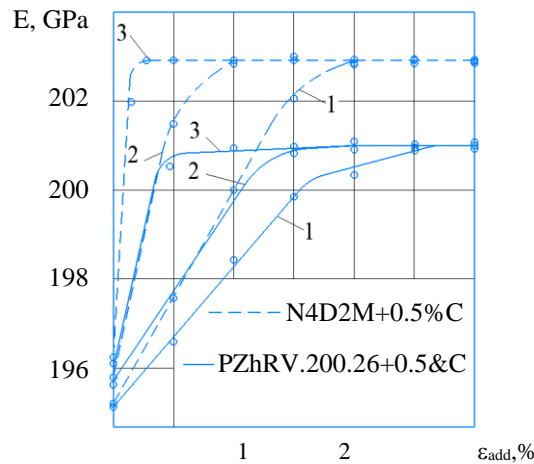


Fig. 8. Dependence of the Young's modulus on the degree of additional plastic deformation of powder steels formed at the temperature of hot post-pressing: 1 — 950 °C, 2 — 1050 °C, 3 — 1150 °C

Based on the conducted studies, we present the values of the critical degree of additional plastic deformation as a function of the technological modes of hot post-pressing (Table 9). The numerator shows data for steel PZhRV2.200.26+0.5 %C, the denominator shows data for N4D2M+0.5%C.

Table 9

Critical degree of deformation at different temperatures of hot pressing

Temperature, 0 C	Initial porosity, %	Critical degree of deformation, %
950	40	2/1.5
	30	2.5/2
	20	3/2
1050	40	1.5/1
	30	1.5/1
	20	2/1.5
1150	40	0.5/0.3
	30	0.5/0.3
	20	1/0.5

Based on the conducted studies, it can be concluded that additional hot plastic deformation contributes to the formation of intracrystalline fusion on the entire contact surface. The addition of graphite to the charge improves the splicing for alloyed iron powder and practically does not affect the use of unalloyed iron powder.

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