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DETERMINING THE INFLUENCE EXERTED BY THE STATIC CONDITIONS OF FINAL SQUEEZING ON THE COMPACTION PROCESS OF IRON-BASED POWDER MATERIALS

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This paper reports a study into the process of re-compaction of powder briquettes in the conditions of static pressing at a pressure of 800 MPa. The technological parameters of the pressing process have been analyzed, which make it possible to improve the compaction of powder briquettes based on iron. Such parameters are the outer greasing, which reduces friction between a green compact and the walls of the press tool matrix, and the firing, which removes the deformation strengthening of the green compacts and increases their plasticity.

The green compacts' sealing mechanism involved in the final squeezing process has been established, which is associated with the grinding of pre-compressed particles due to the strain in the contact areas. The increase in the stressed state of green compacts following the final squeezing was confirmed by the results of studying the residual micro-strains.

The change in the stressed state of iron green compacts has been confirmed by the study into the structurally sensitive characteristics, which include the materials' magnetic and electrical properties. Determining the magnetic characteristics has shown that final squeezing leads to an increase in coercive force, which can be explained by both the increase in the stressed state and the grinding of grains. Investigating the impact exerted by the annealing environment on the value of magnetic characteristics has demonstrated that annealing in hydrogen is more effective in terms of improving magnetic properties than annealing in a vacuum. This is due to the refining of grain boundaries through the processes of reduction of oxide films.

The study of the mechanical characteristics of green compact materials based on iron powder has established that final squeezing leads to an increase in the hardness and strength of materials depending on the conditions of deformation. A significant improvement in the green compacts' strength (820–824 MPa) is due to both a decrease in porosity by 8–10 % and an increase in the contact area as a result of plastic deformation after the annealing

Keywords: final squeezing, coercive force, iron, specific electrical resistance, iron powder, compaction, micro-stresses

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1. Introduction

Among the challenges of our time is the long-projected resource and energy crisis, which requires an adequate organizational and technological response to counteract it. Undoubtedly, it should include strict restrictions on the wide use of expensive and scarce alloying elements and the production of parts with low functional properties. As regards the powder iron metallurgy, this is an improvement in the functional properties of baked products, which is achieved mainly by improving the technology of their manufacture. The latter may include the final squeezing of the resulting powder briquettes; despite the long-standing popularity and seeming triviality of this manufacturing technique, it is not investigated in detail and little used in the practice of powder metallurgy.

The main issue related to making articles by a powder metallurgy method is the residual porosity that reduces the mechanical properties of parts. The use of high-temperature baking modes to overcome this disadvantage leads to a significant volumetric shrinkage of green compacts, which complicates the processes of designing articles of precise geometric dimensions. Therefore, the creation of conditions under which the non-baked green compacts would demonstrate low porosity and, accordingly, small shrinkage when baking, is necessary for the development of energy-saving technologies for the manufacture of articles by a powder metallurgy method, which renders relevance to our work.

2. Literature review and problem statement

One way to increase the density of powder blanks is to use re-deformation operations, which include the static final squeezing, stamping, isostatic and dynamic hot pressing, as shown in works [1, 2]. The method of static hot pressing is a variant of cold static pressing; it actually combines pressing and baking of the workpiece by heating at a temperature above the recrystallization temperature of the powder material [3]. The disadvantages of this method are low performance, as well as low resistance of graphite press equipment, which limits its use in manufacturing. The shortcomings of hot pressing are partially overcome by the technology of SPS (spark plasma sintering) involving the electric-charge or spark-plasma sintering [4]. However, the SPS method is mainly used for processing ceramic materials, which, in the conditions of free baking, have a high residual porosity even with a long time of isothermal aging [5]. The dynamic hot-pressing method is a kind of hot static pressing [6]. Comparing both methods, it should be noted that static hot pressing is dominated by the processes of activated (by external pressure) sintering; in the case of dynamic pressing, it is dominated by the thermomechanical effect on the material. Restrictions on the use of this method in the practice of powder metallurgy are also due to the complexity of the equipment and the inability to manufacture articles of complex shape given a slow increase in pressure. A promising way to deform materials without the use of stamp tools is the free upsetting used for machining multi-ton steel slabs [7]. The disadvantage of upsetting is a significant change in the shape of the workpiece and the deformation heterogeneity, which leads to cracks.

The re-compaction of green compacts in steel matrices makes it possible to improve the mechanical characteristics of finished articles, which is associated with both a decrease in the overall porosity and an increase in the area of interparticle contacts. Double pressing–annealing, given the insufficient strength of steel tools, makes it possible to obtain high density only for a limited range of metal powders; it is not effective enough for the mixtures of iron powder containing large amounts of non-plastic additives. However, the first annealing is also not always acceptable due to the presence of easily oxidized components in the green compact.

The option of overcoming the related difficulties may include warm final squeezing [8] of resulting briquettes, rather than cold; in this case, it is possible not only to eliminate the stratification but also to slightly improve the density. Another known way [9] radically resolves the issue of stratification. It involves the use of a detachable mold of a special design, which makes it possible to adjust the process of pressing a briquette. The application of the specified manufacturing approaches may be acceptable for the primary (before annealing) final squeezing of both the studied and similar heavy-sealed powder mixtures. It should be emphasized that in any case, it is necessary to use iron powders with a high ability to seal, which increases when moving from cold to the warm pressing.

The effectiveness of the process of compaction of powdered materials based on iron during final squeezing may be influenced by various factors that can fundamentally affect the process of green compact deformation and, consequently, the resulting properties of articles. Among the main factors that can be altered to control the compaction process are the plasticity of the material, which is determined by the environment and temperature of annealing, and the chemical composition of the charge (the content of lubricants or solid ingredients) [10]. Other factors determining the sealing of green compacts are the shape and size of the starting particles [11], the degree of wear of the press tool matrix, the temperature of the pressing process, etc. [12].

Of course, increasing the ability of powders to seal, that is, preparatory operations of refining annealing, pumping, acid treatment to change morphology, or alloying by special alloying agents, partially resolves the task to manufacture high-compact powder articles. However, under actual conditions, it is necessary to operate those powder systems that are available, which predetermines the need to develop new combinatorial ways to form high-density powder materials, which include static final squeezing.

3. The aim and objectives of the study

The aim of this work is to determine the impact exerted by the static modes of final squeezing of green compacts based on iron powder on the process of powder material consolidation and the manufacturing of compact articles, which would make it possible to use them in heavy-loaded machine units.

To accomplish the aim, the following tasks have been set:

– to investigate the stressed state of green compacts depending on the conditions of static final squeezing;

– to conduct a metallographic study into the structure of iron green compacts depending on the conditions of static final squeezing;

– to investigate the magnetic, electrical, and mechanical properties of iron green compacts depending on the conditions of final squeezing.

4. Materials and methods to study iron-based green compacts

We used the iron powder of brand PZHRV200.28 delivered as is; pressing it from the state of the stillage was carried out in a separate steel mold with an output working diameter of 10 mm. For a two-side single-stage pressing, we placed the mold with the embedded lower punch on rubber plates in advance, then filled it with batches of the same mass, 8.0 g. Then we pressed the batches and inserted the upper punch, then we carried out pressing (at pressures of 200, 400, 600, and 800 MPa) and unpressed the sample, a cylindrical green compact, according to the methodology given in work [13].

The resulting cylindrical green compacts were subjected to final squeezing in the same mold (Fig. 1). We finally squeezed the briquettes both with and without lubrication; in the latter case, we lubricated the finished briquettes with Vaseline; part of the resulting briquettes, before final squeezing, were annealed in hydrogen at 850 °C for 30 minutes. We trimmed the flash from the briquettes, after which we determined their weight, height, and diameter, based on which we calculated their density and examined their structure, the stressed state, the mechanical, magnetic, and electrical properties.

We conducted a metallographic analysis using the electron scanning microscope SELM-106.

The residual micro-stresses in the green compacts were defined by two methods:

1) a method of speckle interferometry, which implies the relaxation of stresses when drilling a hole [14];

2) a Stokes-Wilson method using the Ultima IY diffractometer software [15].

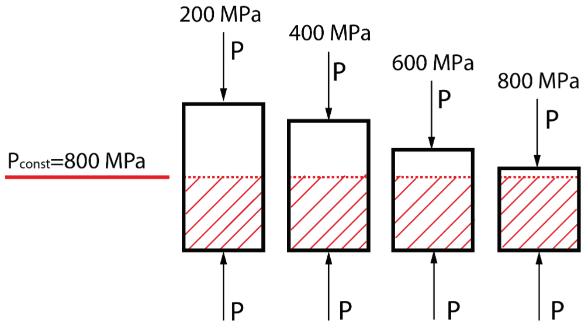


Fig. 1. The schematic of green compacts manufactured at different pressure during primary pressing (200–800 MPa) and after final squeezing at the same pressure of 800 MPa

The value of the residual saturation magnetization was determined by the Millis-Teslameter TPU, whose operation principle is based on the measurement of EMF by the Hall method. We determined coercive force using the coercimeter IKS8-3. The method implies determining the value of the reverse field when reaching zero indication of the registering device during sample demagnetization. We determined the specific electrical resistance of samples at the single-double dc bridge RZOO9, designed for measurement in the range from 10^{-8} to $1.1111 \cdot 10^{8}$ Ω dc in line with TO 3.454.019.

We studied the mechanical characteristics of the resulting composites by determining the strength at compression. The tests involved the calibrated universal machine «CERAMTEST» using cylindrical samples with a load speed of 0.5 mm/min in line with ISO 133314:2011 (E).

The HRB hardness was determined at the device TK-2 using a Rockwell method by pressing a ball into the surface of the sample in line with the requirements of ISO 4498.

5. Results of studying the conditions of final squeezing on the structure, stressed state, and properties of iron-based green compacts

The research results suggest that the annealing contributes to increasing the gain in density at final squeezing much more than the lubrication of green compacts with lubricants (Fig. 2). Thus, final squeezing without annealing leads to an increase in the density gain by only 0.2–0.3 g/cm^3 at a pressure of 600–800 MPa. The annealing in hydrogen at 850 °C ensured a significant increase in density, 0.9–1.0 g/cm^3, at a pressure of 600–800 MPa.

The results of our metallographic analysis carried out with the help of an electron scanning microscope have shown that compaction in the process of final squeezing without annealing occurs due to the additional grinding of pre-compressed particles. This is due to the strain in the places of contact (Fig. 3).

Thus, the structure of green compacts after final squeezing consists of particles measuring 100–150 μm. These particles are surrounded by small particles measuring 30–50 μm, which were formed during the final squeezing as a result of destruction in the contact areas (Fig. 3, *b*).

The increase in the stressed state after final squeezing is confirmed by the results of determining the residual microstrains (Table 1).

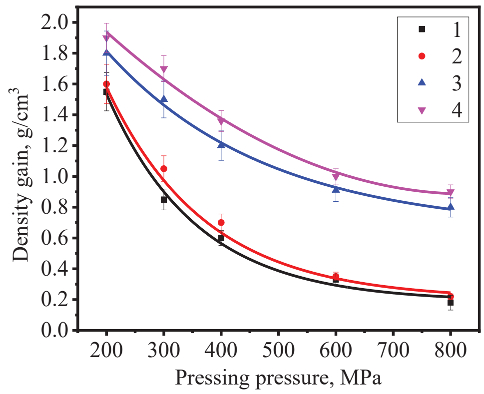
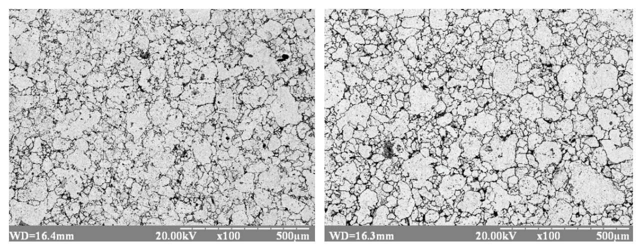


Fig. 2. Gain in the density of briquettes at their final squeezing at 800 MPa under different conditions depending on the starting pressure of their pressing: 1 – without annealing without lubrication; 2 – without annealing with a lubricant; 3 – with annealing without lubrication; 4 – with annealing with a lubricant



a *b*

Fig. 3. The microstructure of iron-based green compacts: *a* – after pressing at 800 MPa; *b* – after pressing and final squeezing at 800 MPa

Table 1

Residual micro-stresses in iron-based green compacts

Treatment type	Residual micro-stresses, MPa	
	Stokes-Wilson method	Speckle interferometry
Pressing at 800 MPa	–220 (±106)	–23–25
Pressing and final squeezing at 800 MPa	–250 (±114)	–28–32
Pressing and annealing at 800 °C	–180 ((±112)	–11–13
Pressing, annealing at 800 °C and final squeezing at 800 MPa	–330 (±165)	–34–42

Characteristics that further determine a change in the stressed state, which are structurally sensitive, are the magnetic and electrical properties of materials, which include, first of all, coercive force and residual magnetization, as well as the value of the specific electrical resistance. The results of our measurement of the specific electrical resistance, coercive force, and residual magnetization are given in Table 2.

We studied the mechanical characteristics of the resulting composites by determining the strength at compression. The tests involved the calibrated universal machine «CERAMTEST» using cylindrical samples with a load speed of 0.5 mm/min in line with ISO 133314:2011 (E).

Table 2

Coercive force, residual magnetization, and specific electrical resistance in iron-based green compacts

Treatment type	Coercive force, A/m	Residual magne-tization, Hs	Specific electrical resistance, μmOhm·cm
Pressing at 800 MPa	400	430	670
Pressing and final squeezing at 800 MPa	480	120	250
Pressing and annealing in hydrogen at 800 °C	316	690	35
Pressing, annealing in hydrogen at 800 °C and final squeezing at 800 MPa	474	230	31
Pressing and annealing in vacuum at 800 °C	390	530	246
Pressing, annealing in vacuum at 800 °C and final squeezing at 800 MPa	474	200	230

6. Discussion of results of studying the effect of final squeezing conditions on the structure, stressed state, and properties of iron-based green compacts

As shown by the results of measuring the residual micro-strains (Table 1), the final squeezing leads to an increase in the stressed state of the material, which is explained by the processes of deformation strengthening and hardening. In this case, the increase in micro-strains is 15–20 % for both methods, which suggests the reliability of our results. The difference in values from different research methods is due to the fact that the method of speckle interferometry determines residual micro-stresses in the volume of the material while and the X-ray method by Stokes-Wilson defines micro-stresses on the surface of the green compact. The annealing of green compacts naturally leads to a relaxation of stresses; the final squeezing after annealing leads to an increase in the stressed state again.

The results of our study into the magnetic characteristics have shown that final squeezing leads to an increase in coercive force (Table 2). This can be due to two reasons: an increase in the stressed state and the grinding of grains (Fig. 3, *b*). The greatest contribution to the change in magnetic properties is from the stressed state of the material, confirmed by the results of measurements of the residual magnetization of green compacts (Table 2), whose value depends primarily on residual stresses after mechanical deformation. Our study of the effect of the annealing environment on the value of magnetic characteristics has shown that annealing in hydrogen is more effective in terms of improving magnetic properties than annealing in a vacuum (Table 2). This is due to the fact that, in addition to the relaxation of stresses, there is the refining of grain boundaries through the processes of oxide film reduction.

Accordingly, the refining of the grain surface leads to an increase in the contact surface, confirmed by the results of measurements of the specific electrical resistance of green compacts (Table 2). Thus, annealing in hydrogen leads to a decrease in the specific electrical resistance by an order of 31–35 μmOhm·cm, which suggests a significant increase in the number of contacts as opposed to annealing in a vacuum.

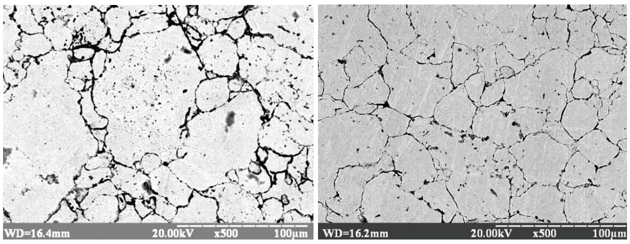
Our study of the mechanical characteristics of green compacts' materials based on iron powder has shown that final squeezing leads to an increase in both hardness and strength (Table 3). Final squeezing without annealing leads to some increase in strength (up to 100 MPa), which is due primarily to the increase of contacts between particles, since porosity, in this case, does not significantly decrease, only by 2–3 %. The annealing before final squeezing provides for a significant increase in the plasticity of the material, which ensures the growth of the strength limit at compression to 820–824 MPa. A significant increase in strength is due to both a decrease in porosity by 8–10 % and an increase in contacts as a result of plastic deformation, which also provides for an increase in hardness to 60–62 HRB (Table 3).

Increasing plasticity before final squeezing is a fundamental factor that determines the effectiveness of the compaction process at final squeezing. To confirm this statement, the final squeezing was carried out after cooling in liquid nitrogen (–196 °C) for 5 minutes. Accordingly, the final squeezing of such green compacts did not provide for a fundamental increase in strength, which grew to only 300 MPa (Table 3), which is less than during final squeezing at room temperature. Cooling in liquid nitrogen leads to a decrease in the plasticity of the material, which is typical of metals with a BCC lattice [16, 17], so the embrittlement of the material led to the grinding of grains, which is confirmed by the results of metallographic analysis (Fig. 4). Thus, the final squeezing of iron-based green compacts after cooling in liquid nitrogen led to a change in the elastic state of the material and the nature of deformation, which caused the embrittlement destruction of the powder particles.

Table 3

The mechanical properties of iron-based green compacts

Treatment type	Hardness, HRB	Yield point, MPa	Compression strength limit, MPa	Relative defor-mation, %
Pressing at 800 MPa	30–32	230–233	270–271	4.25
Pressing and final squeezing at 800 MPa	40–43	342–346	370–372	5.16
Pressing and annealing in hydrogen at 800 °C	20–23	–	–	–
Pressing, annealing in hydrogen at 800 °C and final squeezing at 800 MPa	60–62	350–352	820–824	42.22
Pressing, cooling in liquid nitrogen at – 196 °C, and final squeezing at 800 MPa	48–50	302–306	318–320	1.96



a *b*

Fig. 4. The microstructure of iron-based green compacts: *a* — after pressing at 800 MPa; *b* — after pressing, cooling in liquid nitrogen, and final squeezing at 800 MPa

The reported results allow us to believe that the final squeezing of the annealed briquettes can be considered as a mandatory initial stage of manufacturing operations to obtain powder articles. It should also be noted that the final squeezing of the annealed briquettes brings them into a state of closed porosity. This makes it possible to significantly reduce the requirements for their protection against oxidizing in the annealing process and thereby eliminate the mandatory use of the through-flow protective gas environments. Of course, the limitation of the static process of final squeezing refers to the impossibility of forming powder articles of 5–6 groups of complexity, having many transitions in height relative to the direction of pressure applied. However, given that most structural articles are relatively simple in shape (bushings, rings, washers, valves, etc.), a given method is quite promising in the formation of highly-compacted powder materials, with the ease of implementation. In addition, final squeezing can be effective in compacting heterogeneous powder mixtures that contain not only plastic components but also solid refractory compounds, which may be the subject of subsequent studies.

7. Conclusions

1. We have investigated the process of the final squeezing of powder systems under static pressing conditions and established the main factors affecting the effectiveness of the compaction process of iron green compacts. These include the external lubrication and annealing, which relieves the deformation strengthening and residual stresses in the material from 250 MPa to 180 MPa.

2. It was established that the final squeezing leads to an increase in the strength of green compacts, from 270–271 MPa to 820–824 MPa, which is due primarily to the increase of contacts between the particles, as well as the partial grinding of grains to 30–50 µm.

3. It is shown that the static final squeezing increases the consolidation of iron green compacts, which ensures the formation of a high-density structure and improves the density gain, from 0.2 to 1.2 g/cm^3, already at the pressing stage and, consequently, could reduce the amount of volumetric shrinkage of green compacts at annealing by 10–15 %.

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A STUDY OF THE INFLUENCE PRODUCED BY THE DYNAMICS OF THE WORKING BODIES OF COTTON-PROCESSING MACHINES ON THE COTTON FIBRE QUALITY

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Cotton mass is considered as a compressible porous two-component medium, consisting of a mixture of cotton fibres and air included in the porous medium, which is essential in dynamic treatment processes and requires consideration when planning technological modes.

It was found that the speed of sound in multicomponent media significantly decreases with an increase in the content of the gaseous component. With a certain content of components, it can become less than in each of the components separately. This is due to the fact that with an increase in the content of the gaseous component, the density of the medium increases insignificantly, and the compressibility of air sharply decreases in the pores.

As a result of the research, it was found that the value of the dynamic change in the density of cotton raw materials can significantly exceed its density during static compression. This kind of influence can have both adverse and desirable effects on the primary stage of cotton processing.

The dynamic characteristics of raw cotton as an object of mechanical technology were studied. The values of the speed of sound as a function of the density of cotton raw materials were determined on the basis of the theory of a two-component porous medium. The types of the dynamic compression curve of raw cotton have been established. Experimental studies on the compressibility of raw cotton are generalized.

From the analysis of the cleaning processing of fibres and seeds on cleaning machines, it follows that when assigning a technological processing mode, it is necessary to comply it with the value of the sound speed for a given density of raw materials. It is necessary to avoid such rates of penetration of the working bodies into raw materials that are commensurate with the speed of sound at a given raw material density. This local dramatic increase in cotton media characteristics is a significant cause of fibre damage

Keywords: fibrous material, multicomponent medium, speed of sound, dynamic processes, layer deformation

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1. Introduction

One of the fundamental characteristics of a fibrous material that determines its dynamic behaviour is the speed of sound propagation in it. The magnitude of the speed of sound determines the boundary at which the phenomenon of compressibility of the processed cotton medium begins to manifest itself while the smoothness, pressure and temperature in it significantly increase. This effect can adversely influence the damage to cotton fibres but in some cases can be positive.

Research on the speed of sound can result in its direct practical application in matters of cleaning technology by vibration and wave methods. The need to develop and implement such methods is due to the following.

The existing mechanical and technological processes for the primary processing of raw cotton – cleaning from fine impurities, ginning (saw and roller), fibre cleaning, in-plant and