Structure and properties of hot-deformed powder steels microalloyed by aluminium

V Y Dorofeyev, A N Sviridova, Y M Berezhnoy, E N Bessarabov, K S Kochkarova, V N Pustovoit and S V Sviridova

1 Platov South-Russian State Polytechnic University (NPI), 132 Prosveshcheniya str, Novocherkassk, Rostov oblast’, 346428 Russia
2 North-Caucasian State Academy, 36 Stavropol’skaya str, Cherkessk, KChR, 369000 Russia
3 Don State Technical University, 1 Gagarin sq., Rostov-on-Don, 344000 Russia
4 Derzhavin Tambov State University, 33 Internatsional’naya str., Tambov, 392036, Russia

5 E-mail: dvuy56.56@mail.ru

Abstract: The feasibility of cohesion degree enhancement, mechanical properties, and contact durability of powder forged steels due to microalloying with aluminium was studied. Unalloyed iron powders with various impurity contents, as well as atomized powder of low-alloy chromium-molybdenum steel, were used as the basis for the producing mixtures. Aluminum was doped as ferroaluminum. Specimens for mechanical and fatigue tests were produced by powder forging. Post-heat treatment was carried out according to two flow routes. The first flow route included carburizing, hardening and low tempering. The second flow route corresponded to high-temperature thermomechanical processing, which was carried out immediately after the completion of hot pressing porous preform. It was found that doping aluminum microadditives in hot-deformed powder steels provides the feasibility of increasing bending strength, impact strength and fatigue life under contact or low-cycle loading. This is associated with an enhancement of the conditions for interparticle joining during hot forging due to the generation of interlayers of ferrite in the interparticle zones as well as to the activation of cohesive interactions as a result of accelerating the process of dynamic recrystallization

1. Introduction

An increase in mechanical and operational properties of materials and products based on them necessitates the development of effective methods to prevent brittle intercrystalline fracture. This problem is of particular relevance in relation to steels obtained from metal powders using methods of hot pressure treatment [1–3]. Steels of this type contain an increased amount of impurities in comparison with cast steels, which is due to the peculiarities of technology for the production of initial powders. In addition, the short duration of deformation action on the material during hot pressing porous preform does not always provide the possibility of forming cohesive bonds at the boundaries between powder particles, i.e., joining [5, 6]. In accordance with known concepts, joining is a dynamic recrystallization process that proceeds simultaneously with the deformation of material of porous preform. The result of this process is the generation of common grains at the interparticle boundaries.
Doping microadditives of alloying elements is a promising method of directed action on the formation of structure of interparticle boundaries and the activation of joining processes. Doping calcium- or sodium-bearing microadditives to iron-based powder blends provides the feasibility of reducing oxides of alloying elements, improving the conditions of homogenization and sintering as well as contact interaction [7–9].

In addition to alkali and alkaline-earth metals, aluminum is a promising microalloying element in this context. Being bound to nitride, aluminum inhibits the growth of austenite grain upon heating and promotes the generation of a fine-grained steel structure [10]. In contrast, aluminum dissolved in a solid solution causes an increase in austenite grain upon heating due to an increase in the free energy of grain boundaries [11]. The latter circumstance in the application to the production of cast steels is negative in nature, as it causes a decrease in strength indicators. However, when considering the features of the processes of formation of structure and properties of powder steels obtained using hot forging porous preforms (HFPP), it seems promising. This is due to the feasibility of accelerating the migration of interparticle surfaces during the performance of technological operations of HFPP and the generation of common recrystallized grains at interparticle boundaries, which seems to be especially significant when producing alloyed powder steels. It was shown that doping additional alloying elements increases the values of the work of segregation retardation $A_{sr}$ [12]. In particular, the values for compositions based on chromium-molybdenum and unalloyed iron powder are $(\times 10^{-5} \text{erg/sm}^2)$ 4.43 and 1.48, respectively.

Since the feasibility of using powder steels in the production of heavily loaded parts is associated with the need for alloying, it becomes obvious the feasibility of research aimed at developing effective ways to activate the processes of interparticle cohesion and to reduce the tendency to develop brittle fracture of alloyed powder steels.

Microalloying powder steels with aluminum is associated with the need to solve several problems. First of all, it is necessary to ensure the distribution of aluminum in the surface layers of iron powder particles, while not allowing a substantial migration of aluminum atoms deep into the iron particles. The surface of iron powder particles is coated with a layer of iron oxides. The thickness of oxide layer on the surface of chromium-molybdenum iron powder particles is $\sim 6–7$ nm [13]. In this connection it becomes obvious that another problem needs to be solved. The problem is the reduction of the oxide layer, which is a barrier to diffusion of aluminum atoms.

Brittle fracture can develop under different types of loading. The most critical are dynamic, fatigue and contact loads [14, 15]. One of the main characteristics that determine the mechanism of powder materials’ failure is the relative density ($\rho_r$). At $\rho_r \sim 0.9$ the strength and fracture toughness of steels are symbatic and proportional to the size of sinter necks, which is a significant difference from cast steels, for which these characteristics are antagonistic [16]. In hot-deformed powder steels (at $\rho_r \sim 0.99$), as well as in cast steels, an increase in strength above 1000 MPa causes a change in the viscous fracture mechanism to a low-energetic cleavage. In this case, the presence of small amount of retained austenite is considered as a positive factor. A similar conclusion was made when studying the influence of retained austenite and residual compressive stresses on the contact durability of cast carburizing steel AISI 8620. Samples with higher residual compressive stresses and retained austenite content showed higher contact endurance [17].

In sintered steels at $\rho_r \leq 0.9$ the occurrence of local plastic deformations accelerates the initiation of fatigue cracks in the subsurface layers, and therefore the existence of retained austenite is considered undesirable [18]. Nevertheless, the generation of an inhomogeneous structure with zones of Ni-enriched austenite surrounded by a martensitic shell provides the feasibility of inhibiting the development of surface crack [19]. Under action of contact loads the retained austenite in the carburized layer can transform into martensite. However, this process does not complete until the end [20]. The question of the influence of retained austenite on the contact durability of powder steels does not have a definite answer. The type of structural components, their ratio and distribution in the volume of the material are the determining factors in this regard.
Mode and conditions of final heat treatment also have a significant impact on the mechanical and operational properties of steels [21]. Powder steels are characterized by reduced hardenability, which is associated with the presence of a large number of nonmetallic inclusions, which are crystallization centers and accelerate the decomposition of austenite on cooling [22]. An effective method to increase the resistance of steels to the development of brittle fracture is thermomechanical processing. The performance of high-temperature thermomechanical processing (HTTMP) provides the feasibility of a simultaneous gain in strength and ductility [23]. In this case, a slight increasing the amount of retained austenite is possible [24]. The specifics of producing hot-deformed powder steels (HDPS) processing suggests the feasibility of implementing HTTMP immediately after completion of hot repressing blank [2].

The purpose of this work is to study the feasibility of increasing mechanical properties and contact durability of HDPS in the hardened condition or in the condition after thermomechanical treatment at the expense of microalloying with aluminum.

2. Experimental Procedure

This work is a continuation of a set of studies published earlier [9, 25]. In this regard, to ensure the possibility of a comparative analysis, the composition of base iron powders was identical to those used previously. Iron powders PZhV2.160.26 manufactured by Sulin Metallurgical Plant, as well as AstaloyCrM and ABC100.30 manufactured by Höganäs AB were used. Figure 1 shows the flow diagram for the production of HDPS. Carbon was doped as pencil graphite powder GK-1 GOST 4404-78 in an amount that ensured the production of eutectoid steels. Milled ferroaluminum FA-50 was used as a microalloying additive. The average size of particles measures ≤ 63 μm. Planetary centrifugal mill SAND-1 was used when mixing powders of iron, ferroaluminum and graphite. To compensate for the cooling surface layers of preform the die was heated to $T_D = 600 \, ^\circ C$.

![Figure 1. The flow diagram of specimen production.](image)

Flow route 1 provided for the carburization and subsequent heat treatment of hot forged samples. Carburization was carried out to compensate for decarburization of surface layer. Flow route 2 provided for performance of thermal hardening operations immediately after hot deformation of preform.
(HTTMP). Since the possibility of carburization was excluded, the decarburized surface layer was removed by machining.

Prismatic samples of 10×10×55 mm and 5×10×55 mm in size and cylindrical samples of ø 26 × 6 mm in size were obtained. The procedure for determining the mechanical properties and contact endurance of powder steels was consistent with that described in [9]. Contact endurance was characterized by the lifetime $N_{90}$, expressed in hours and corresponding to the probability of failure of 90% of the samples [26].

Fatigue life was determined using prismatic samples of the size 5×10×55 mm under conditions of low-cycle rigid loading according to the procedure [27]. As a characteristic of low-cycle fatigue life, the number of cycles until the failure of sample ($N_{lcf}$) was used.

Metallographic investigations were performed using an AltamiMET-1M optical microscope (AltamiLtd., Russia) on etched and unetched slices. Etching was performed in 3% nital. Fractures of samples were studied using a Quanta 200 i 3D scanning microanalyzer.

3. Results and Discussion
Microalloying powder steels with aluminum caused a change in the structure of interparticle zones. In porous cold-pressed blanks ferroaluminum particles are observed in the form of separate isolated inclusions localized at the boundaries between particles of iron powder (figure 2, a). At the initial stage of sintering ferroaluminum particles melt, forming “islands” in the interparticle zones (figure 2, b). Thin film, coating the particles of iron powder, is formed at a later time.

![Figure 2. Structure of powder steel with aluminum microadditives. As-cold-pressed (a); as-sintered (b); as-forged (c). Base powder PZhV2.160.26. $C_{Al} = 0.4$ wt. %](image)

![Figure 3. Contact durability of hot-deformed powder steels versus $C_{Al}$. Base powder: AstaloyCrM (1); ABC100.30 (2); PZhV2.160.26 (3). $\tau_s = 30$ min. Flow route: 1 (a); 2 (b).](image)
The activation energy of diffusion of carbon and aluminum in iron comprises, respectively, 118.5 and 184.2 J/mol, which determines higher diffusion activity of carbon in iron compared with aluminum [28]. In addition, aluminum is a graphitizing element and slows down the dissolution of carbon in iron [29]. In this connection diffusion of carbon from the composition of graphite particles into the depth of iron particles occurs at the initial stage of sintering. As this takes place, aluminum, localized near interparticle boundaries, promotes the generation of ferrite interlayers. The structure of sintered specimens, the sintering duration of which ($\tau_s$) did not exceed 30 min, is fine-grained pearlite (grade 3–4 on the scale number 2 GOST 8233-56) with ferrite interlayers. At $\tau_s > 30$ min homogenization proceeds and structural heterogeneity decreases.

The above-mentioned features of structural components' distribution in as-sintered powder steels had a decisive influence on the generation of structure and properties during HFPP. Figure 3 shows the aluminum content ($C_{Al}$) dependences of the contact durability of powder steels. Dependencies are extreme. As $C_{Al}$ values increase up to 0.4 wt. % values of $N_{90}$ increase and reach a maximum. This is due to the enhancement of conditions of contact interaction between iron powder particles during hot deformation, which is caused by the existence of interlayers of ferrite in the interparticle zones formed during sintering. Decarburization of contact surfaces helps to reduce the optimum temperature of hammer welding steels [30]. In addition, the feasibility of manifestation of another mechanism should also be taken into account. Aluminum, which is part of a solid solution in iron and is localized in the interparticle zones, activates the process of dynamic recrystallization, as a result of which common grains are formed at the interparticle boundaries (figure 2, c). On further increase in $C_{Al}$ over 0.4 wt. % values of $N_{90}$ fall due to an increased risk of formation of inclusions of intermetallic compounds $Fe_2Al_5$ and $FeAl_3$, which embrittles interparticle zones (figure 4, a).

![Figure 4](image1)

**Figure 4.** The fracture surface of the samples of HDPS. Base powder PZhV2.160.26. Flow route 1. $C_{Al} = 0.6$ (a); 0 (b); 0.4 wt. % (c). After contact (a) and low-cycle fatigue tests (b and c).

Doping aluminum microadditives prevents the creation of carbide net in the surface layer of forged specimens during carburizing (grade 1 on the scale number 4 GOST 801-78). In contrast, in the structure of specimens-witnesses without aluminum microadditives the residues of carbide network are observed and correspond to grades 2–3. The performance of carburizing, heat or thermomechanical treatment (flow routes 1 and 2) caused diffusion redistribution of carbon in the volume of samples and transformation of ferrite interlayers into bainite areas (HV 400–450), which retained localization in the vicinity of former interparticle boundaries. Regardless of the type of source iron powder the base structure of specimens, produced according flow route 1, is medium-needle martensite (grade 5 on the scale number 3 GOST 8233-56; HV 700–720) and retained austenite (HV 450–500). In the carburized layer of specimens made from powders AstaloyCrM and ABC100,30 fine needle-type martensite (grade
4 on the scale number 3 GOST 8233-56; HV 730–740) and retained austenite (HV 520–570) are observed, and bainite areas are not observed (in contrast to samples based on PZhV2.160.26).

The performance of HTTMP determined gain in the dispersiveness of the structure partials of powder steels. The structure of specimens made from powders AstaloyCrM and ABC100.30 after HTTMP (flow route 2, the decarburized layer was removed during machining) comprises a very fine needle-type martensite (grade 2 on the scale number 3 GOST 8233-56; HV 770–800) and retained austenite (HV 500–530) with zones of bainite. In the structure of specimens made from powder PZhV2.160.26 with a relatively high contaminant load fine needle-type martensite (grade 4 on the scale number 3 GOST 8233-56; HV 710–730) and retained austenite (HV 470–490) are observed.

Despite the more dispersed character of martensite, the values of $N_{90}$ of samples after HTTMP turned out to be lower than those of heat-treated samples (compare plots in figures 3, b and 3, a), which is due to the existence of structural heterogeneity regions. Regardless of the technological background, the highest contact durability was shown by specimens made from atomized chromium-molybdenum powder, and the smallest one was shown by specimens based on reduced powder PZhV2.160.26 with a high contaminant load (plots 1 and 3 in figures 3, a and 3, b).

![Figure 5. Mechanical properties of hot-deformed powder steels versus $\tau_s$. Base powder: AstaloyCrM (1, 4, 7); ABC100.30 (2, 5, 8); PZhV2.160.26 (3, 6, 9). BS (1, 2, 3); $N_{lcf}$ (4, 5, 6); IS (7, 8, 9). Flow route: 1 (a); 2 (b). $C_{Al} = 0.4$ wt. %.

Since distribution of aluminum has a decisive effect on the generation of structure of interparticle zones of microalloyed steels, this parameter was optimized in this work. The duration of vacuum sintering ($\tau_s$) was chosen as the control parameter. The $\tau_s$-dependences of bending strength (BS) are monotonic. As $\tau_s$ values are increased bending strength increases. However, such increasing is not significant (figure 5, plots 1–3). As this takes place, at $\tau_s \geq 30 \text{ min}$ it practically ceases, which is due to a weak sensitivity of the strength indicators to cohesion degree [6]. The values of BS of heat-treated samples in all cases turned out to be higher in comparison with samples after HTTMP (compare plots in figures 5, a and 5, b), which is associated with the existence of a carbide surface texture in heat-treated samples. The highest BS values were shown by specimens produced on the basis of powder AstaloyCrM, which is associated with the strengthening influence of chromium and molybdenum (plots 1). The strength of carbon steels based on powders ABC100.30 and PZhV2.160.26 is somewhat lower, and the smallest is observed for samples made from powder PZhV2.160.26 with a high contaminants load (compare plots 2 and 3).

Compared to BS, the characteristics of fatigue life under low-cycle loading ($N_{lcf}$) and impact toughness (IS) are largely caused by cohesion degree and dispersiveness of structural components. In this connection values of $N_{lcf}$ of specimens after HTTMP turned out to be higher than the corresponding values of heat-treated specimens (compare plots 4–6 and 7–9 in figures 5, a and 5, b), which is associated
with the generation of a very fine needle-type (AstaloyCrM and ABC100.30) or fine needle-type (PZhV2.160.26) martensite during HTTMP (see above). Obviously in this case the existence of bainite in zones of former interparticle boundaries is not a softening factor. In this regard, the following assumption can be made. It is known that brittle fracture can develop in a nominally plastic phase on condition that the difference in yield strengths of structural components is significant. The brittle fracture of steel with a martensite structure in the presence of a fine ferrite network around the martensitic areas develops along the plastic component (ferrite) [31]. As this takes place the same stress state is generated in ferrite as in notch. Since the microhardness of bainite in powder microalloyed steels was in the range from 400 to 450 HV, this embrittlement condition was not met.

Contrary to bending strength and contact durability the highest impact strength and fatigue life under low-cycle loading both in heat-treated condition (flow route 1) and in condition after HTTMP (flow route 2) were demonstrated by samples based on powder ABC100.30 with a low content of impurities. Samples based on powder AstaloyCrM have slightly lower values of the indicated characteristics, and the samples made from powder PZhV2.160.26 with a high content of impurities have the smallest.

When studying fracture surfaces of samples destroyed during low-cycle fatigue tests, a crack development zone, a transition zone, and a final fracture zone are observed. Crack nucleates near nonmetallic precipitates, which is clearly seen in fractures of specimens based on powder PZhV2.160.26. Doping aluminum microadditives influenced on the kinetics of fatigue failure in all these zones. In zone of crack development the fracture of microalloyed samples has a more developed relief, indicating a higher energy capacity of fracture compared with samples-witnesses (figure 4, b and 4, c). The transition zone of fracture of specimens-witnesses is mainly intercrystalline in nature, in contrast to microalloyed samples, which are characterized by transcryalline cleavage, indicating satisfactory cohesion between the particles of iron powder. Final fracture zone of microalloyed samples and specimens-witnesses has a woolly character. As this takes place a significant number of cracks and delaminations are observed in samples-witnesses.

4. Conclusions
Microalloying hot-forged powder steels with aluminum provides the opportunity to increase bending strength, impact strength and fatigue life under conditions of contact or low-cycle loading, which is associated with improved conditions for interparticle joining during hot forging porous preforms at the expense of generation of ferrite interlayers in the interparticle zones and the activation of cohesive interaction as a result of accelerating process of dynamic recrystallization and generation of common grains at interparticle boundaries.

The performance of carburizing and heat treatment of hot-forged powder steels provides higher values of bending strength and contact endurance as compared to HTTMP. To achieve higher impact strength and low-cycle fatigue life, it is advisable to use HTTMP, which causes the formation of finely dispersed structures.

Enhancement of cohesion degree in HDPS with doping aluminum microadditives provides increasing the proportion of transcryalline component in the fracture of microalloyed samples in comparison with samples-witnesses.

The maximum values \((N_{90} \approx 560 \text{ h}; BS \approx 3900 \text{ MPa})\) of contact endurance and bending strength were demonstrated by samples with microadditive 0.4 wt. % of aluminum, obtained on the basis of powder AstaloyCrM in the condition after carburizing and heat treatment. Steels based on powder ABC100.30 in the condition after HTTMP display maximum impact strength and low-cycle fatigue life.

Acknowledgments
This work was carried out with the financial support of the Russian Foundation for Basic Research, grant No 19-08-00107 A. The images on a scanning microscope-microanalyzer Quanta 200 i 3D were obtained at the Nanotechnology Center for Collective Use of the Platov South-Russian State Polytechnic University (NPI). Authors are grateful to Höganäs East Europe Ltd. for the provided iron powders manufactured by Höganäs AB.
References

[1] Kuhn H A and Ferguson B L 1990 Powder Forging (Princeton, New Jersey: MPIF) p 270

[2] Dorofeyev Y G 1977 Dynamic Hot Pressing Powder Blanks (Moscow: Metallurgy Press) p 216

[3] Salak A, Ivanov V S, Veles P and Selecka M 1988 Hutnicke Listy 43 399-405

[4] Selecka M and Salak A 1999 Wear 236 47-54

[5] Dorofeev Y G 1970 Soviet Powder Metallurgy and Metal Ceramics 9 809-12

[6] Dorofeev Y G and Popov S N 1971 Soviet Powder Metallurgy and Metal Ceramics 10 118-24

[7] D’yachkova L N and Kerzhentseva L F 2012 University News Powder Metallurgy and Functional Coatings 4 32-7

[8] Dyachkova L N and Dechko M M 2016 Russian Journal of Non-Ferrous Metals 57(5) 477-83

[9] Dorofeyev V Y, Sviridova A N, Berezhnoy Y M, Bessarabov E N, Kochkarova K S and Tamadaev V G 2019 IOP Conf. Ser.: Mater. Sci. Eng. 537 022046

[10] Samsonov G V, Kulik O P and Polischuk V S 1978 Obtaining and Methods for the Analysis of Nitrides (Kiev: Naukova Dumka Press) p 320

[11] Brown M P 1982 Microalloying Steel (Kiev: Naukova Dumka Press) p 303

[12] Dorofeyev V Y and Eygorov S N 2005 Science of Sintering 37(3) 225-30

[13] Karlsson H, Nyborg L and Berg S 2005 Powder Metallurgy 48(1) 51-8

[14] Rittel D 2014 Dynamic testing of materials: selected topics Constitutive Relations under Impact Loadings (CISM International Centre for Mechanical Sciences 552 59-86

[15] Rudenko S P and Val’ko A L 2014 Contact Fatigue of Gears of Transmissions of Energy-Efficient Machines (Minsk: Belarusian Science Publishing House) p 126

[16] Phillips R A, King J E and Moon J R 2000 Fracture toughness of some high density PM steels Powder Metallurgy 43(1) 43-8

[17] Shen Y, Moghadam S M, Sadeghi F, Paulson K and Trice R W 2015 Effect of retained austenite – Compressive residual stresses on rolling contact fatigue life of carburized AISI 8620 steel International Journal of Fatigue 75 135-44

[18] Mekonone S T, Pahl W and Molinari A 2018 Powder Metallurgy 61(3) 187-96

[19] Bergmark A and Alzati L 2005 Fatigue Fract Engng Mater Struct 28 229-35

[20] Sauermann I and Beiss P Proc. European Congress and Exhibition on Powder Metallurgy Euro PM2007 (Toulouse) vol 1 (European Powder Metallurgy Association) pp 115-22

[21] Bocchini G Proc. European Congress and Exhibition on Powder Metallurgy Euro PM2009 (Copenhagen) 1 pp 107-14

[22] Antsiferov V N and Cherepanova T G 1981 Structure of Sintered Steels (Moscow: Metallurgy Press) p 110

[23] Bershstein M L, Zaîmovskî V A and Kaputkina L M 1983 Thermomechanical Processing of Steel (Moscow: Metallurgy Press) p 480

[24] Tonysheva O A, Voznesenskaya N M, Shestakov I I and Eliseev E A 2017 The influence of high-temperature thermostatic treatment on the structure and properties of high-strength corrosion-resistant austenitic-martensitic steel 17Kh13N4K6SAM3ch Aviation Materials and Technology 46(1) 11-6

[25] Dorofeyev V Y, Sviridova A N and Svistun L I 2019 The effect of sodium microalloying on the rolling contact fatigue and mechanical properties of hot-deformed powder steels University News Powder metallurgy and functional coatings 4 4-13

[26] Orlov A V, Chernenskii O N and Nesterov V M 1980 Tests of structural materials for contact fatigue (Moscow: Mechanical Engineering Press) p 110

[27] Sarbash R I 1988 Fatigue endurance of specimens of a powder steel in low-cycle hard loading conditions Soviet Powder Metallurgy and Metal Ceramics 27(9) 746-9

[28] Getsriken S D and Dekhtyar I Y 1960 Solid Phase Diffusion in Metals and Alloys (Moscow: Physical and Mathematical Literature State Press) p 556

[29] Sherman A D et al 1991 Cast Iron Reference Edition ed A D Sherman and A A Zhukov (Moscow: Metallurgy Press) p 576
[30] Surovtsev A P and Mytsik A P 1988 Thermodynamic evaluation of the solid phase weldability of iron-carbon alloys *Physics and Chemistry of Materials Treatment* **22**(1) 83-6

[31] Sarrak V I and Filippov G A 1974 Brittleness of martensite *Metal Science and Heat Treatment* **20**(4) 279-85