Effect of microalloying on the stability of the endurance characteristics of hot-deformed powder steels

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Abstract. The stability of the characteristics of the contact and low-cycle fatigue life of steels obtained from iron powders with different contents of alloying and microalloying elements, as well as impurities, has been studied. The carbon content in powder steels was varied. It has been established that doping Na or Ca microadditives contributes to an increase in the stability and absolute value of the parameters of the contact and low-cycle fatigue life of hot-deformed powder steels due to the activation of cohesive interaction on interparticle surfaces and carbon diffusion, which contributes to the formation of a homogeneous structure. Doping microadditives of Al contributes to an increase in the contact and low-cycle fatigue life of the powder steels in comparison with the samples-witnesses. However, the formation of zones of structural heterogeneity in steels containing more than 0.4 wt. % C, causes a decrease in the stability of these characteristics.

1. Introduction

The stability of the mechanical properties of materials largely makes it possible their use in manufacturing parts for various purposes. The importance of this factor increases in the case of manufacturing products operating at low temperatures and exposed to dynamic, fatigue, static and wear loads. Brittle materials (ceramics, sitalls, glass, cast iron, etc.) are characterized by low stability of mechanical properties, which is associated with the heterogeneity and defectiveness of the structure caused by the instability of technological processes of their preparation [1].

The cause for the instability of the compact steel’s properties can be the presence of internal stresses and areas of metastable austenite. In this regard, heat treatment is an effective method for increasing the stability indicators of such steels. As an additional heat treatment of high-strength strip steel, it is recommended to use artificial aging, which provides a significant decrease in the value of internal stresses and promotes the precipitation of dispersed carbides and carbonitrides that stabilize dislocations [2].

The influence of technological modes of deformation processing, test conditions, as well as alloying on the mechanical properties of metastable austenitic steels and indicators of their stability is...
often ambiguous. In particular, if the crack resistance decreases with the introduction of alloying elements, then the indicators of its stability increase. On the contrary, an increase in the degree of deformation leads to an increase in crack resistance and a decrease in its stability [3]. An objective criterion for the stability of austenite and the mechanical properties of steels of this class is the coefficient characterizing the degree of decomposition of austenite.

Powder steels are characterized by low stability of mechanical properties, which is associated with the presence of a large number of impurities, chemical and structural inhomogeneities, discontinuities and defects of various origins (pores, microcracks, delamination, etc.). Indicative in this respect is the diagram, which shows the change in the scatter of the values of the nickel content in powders of the Distaloy type at different time stages of their production, given in [4]. This diagram clearly illustrates the fact that the development of technologies for obtaining Distaloy powders was aimed at reducing the spread of nickel content values. This made it possible to significantly reduce the instability of the values of the mechanical properties of the materials obtained, as well as the dimensions and characteristics of the shape and the relative position of the surfaces of products.

The instability of the sizes and properties of powder materials and products is caused by the segregation of the components of the initial mixtures during their preparation [5]. Each technological operation of obtaining a powder product has a hereditary effect both on the absolute values of the characteristics of mechanical properties and dimensional accuracy of the products obtained, and on the indicators of their stability [6].

The cause for the instability of the sizes and properties of powder materials and products based on them may be the unsatisfactory quality of interparticle jointing. With regard to powder materials obtained by pressing-sintering technology, this leads to uneven localization of the setting bridges, which depends on the direction of the acting forces during cold pressing and causes anisotropy of shrinkage during sintering [7, 8].

The instability of the properties of hot-deformed powder steels (HDPS) is often associated with the unsatisfactory quality of interparticle jointing, which is formed at the stage of repressing of the heated porous workpiece in the die matrix. Earlier it was shown that the introduction of microadditives of alloying elements provides an increase in the absolute value and stability of the values of the mechanical properties of HDPS by improving the quality of interparticle jointing [9]. The stability of the mechanical properties of steels based on unalloyed iron powders was studied in relation to the characteristics of impact toughness and crack resistance. Determination of the impact strength implies the possibility of using smooth samples and does not cause difficulties. On the contrary, the assessment of crack resistance is associated with the need to apply a stress concentrator, which is not always possible. In particular, when testing samples with a hardened surface layer, the application of a notch will lead to an incorrect estimate of the value of crack resistance.

Various types of fatigue tests are widely used to assess the performance of materials under cyclic loading [10]. However, the currently available information on the fatigue characteristics of powder steels is not systematized, despite the large amount of experimental data obtained. This does not allow using it in the well-known software package nCode based on Ansys in order to predict the resource of powder materials and products. As applied to powder materials obtained by hot forging of porous blanks, this problem is of particular importance due to the low reproducibility of properties.

In addition, the study of the behavior of a hot-deformed powder material under the influence of cyclic loads is of scientific importance. This is due to the fact that a significant part of the research carried out to date has been devoted to the study of the dependence of the fatigue characteristics of powder steels on density. The upper limit of the studied values of the density of powder steels (ρ), as a rule, was limited to the values (7.2 - 7.4) · 10^3 kg / m^3. The number of studies devoted to the study of the endurance characteristics of powder steels in a high-density state is significantly less. This indicates the feasibility of further research on HDPS endurance. The availability of relevant information will provide the possibility of plotting the dependences of the fatigue strength of powder steels on the density in a wider range of its values. In this case, the obtained values of HDPS density and properties can be (with certain adjustments caused by the presence of impurities and structural
features) a standard. In particular, in the German-language literature P. Beiss proposed a generalized formula of M. Yu. Bal'shin:

\[ P = P_0 \left( \frac{\rho}{\rho_0} \right)^m, \]

where: \( P \) – material property with porosity; \( P_0 \) – property of a compact material; \( \rho \) – density after sintering; \( \rho_0 \) – density of the compact material; \( m \) – exponential multiplier.

The problem lies in determining the values of \( P_0 \) and \( \rho_0 \). The well-known technique for \( \rho_0 \) determining from the volume fraction of constituent components does not take into account changes in density due to diffusion, phase and structural transformations. The method for \( \rho_0 \) determining, which provides for the melting of powder molding, also looks not quite correct. The liquation of the melt components, the formation of slag on the surface of the melt, the occurrence of crystallization, phase and structural transformations during its cooling may have a significant effect on the \( \rho_0 \) values and the properties of such a reference material.

This work is a continuation of research of the HDPS structure and properties with microadditions of alloying elements [11]. Its purpose is to study the possibility of increasing the stability of the HDPS endurance characteristics by introducing microadditions of alloying elements.

2. Research methods

For fatigue tests, samples were made on the basis of PZhV2.160.26 iron powder manufactured by the Sulinsky metallurgical plant, as well as AstaloyCrM chromium-molybdenum iron powder and ABC100.30 unalloyed iron powder manufactured by HöganäsAB (Sweden). The chemical composition of iron powders is given in research [11]. The technological scheme for obtaining samples is shown in figure 1.

The carbon content in powder steels (\( C_{Cm} \)) was varied. Carbon was introduced in the form of pencil graphite powder GK-1 GOST 4404-78. Sodium was introduced in the form of bicarbonate NaHCO\(_3\) GOST 2156-76, calcium in the form of carbonate CaCO\(_3\) GOST 4530-76. During microalloying with aluminum powder of crushed ferroaluminium FA-50 TU U 27.3 - 13533123 - 001 - 20 fraction (-0.063) mm was used. The content of microadditives corresponded to the optimal values established earlier: \( C_{Na} = 0.2 \text{ wt. \%}; C_{Ca} = 0.3 \text{ wt. \%}; C_{Al} = 0.4 \text{ wt. \%}. \)

To determine the characteristics of endurance of powder steels and carry out structural analysis, prismatic samples with a size of 5 × 10 × 55 mm, as well as cylindrical samples with a size of \( \varnothing \) 26 × 6 mm, were obtained. To compensate for the cooling of the surface layers of the workpiece, hot forging was carried out in a mold, the heating temperature of which was \( T_D = 600 ^\circ C \). Hot-forged samples were carburized to compensate for decarburization, followed by heat treatment. To reduce the risk of the delayed destruction, the release duration was increased to the usually recommended values and was 3 hours.

The method for determining the characteristics of contact (\( N_{90} \)) and low-cycle fatigue life (\( N_{cr} \)) corresponded to that described earlier [11, 12]. The stability of mechanical properties was assessed by the value of the coefficient of variation [13]

\[ V = \frac{s}{\bar{X}}, \]

where \( \bar{X} = \frac{1}{n} \sum n X_i \) – arithmetic mean value; \( n = 10 \) – number of experiments; \( X_i \) – absolute value of a mechanical property in the experiment; \( S = \sqrt{D} \) – standard deviation; \( D = \frac{(X_i - \bar{X})^2}{\sum n} \) – dispersion.
Figure 1. The flow diagram of sample production.

Metallographic analysis was carried out using an AltamiMET-1M optical microscope (Altami LLC, Russia) on etched (3% Nital) and non-etched thin sections. The fractures of the samples were studied using a Quanta 200 i 3D scanning microscope-microanalyzer.

3. Results and discussion

Figure 2 shows the dependences of the endurance characteristics of microalloyed powder steels and their stability on the carbon content. The $N_{90}$, $N_{lcf}$ ($C_{cm}$) dependences for steels with microadditives of calcium were published earlier. The indicated dependences are non-monotonic. An increase in the $C_{cm}$ values to 0.8 wt. % contributes to an increase in the characteristics of endurance, which is associated with an increase in the amount of martensite in the structure of steels. With a further increase in the $C_{cm}$ value, the $N_{90}$ and $N_{lcf}$ values decrease due to the precipitation of excess cementite.

The largest values of $N_{90}$ are observed on samples of chromium-molybdenum steel of eutectoid composition with a microaddition of Na (figure 2, a; curve 6). The samples based on undoped iron powder ABC100.30 showed slightly lower $N_{90}$ values (curve 5). The lowest contact endurance was demonstrated by reference samples based on PZhV2.160.26 powder without microadditions of alloying elements (curve 8). Microalloying made it possible to increase the contact endurance of the samples obtained on the basis of this powder, despite the relatively high number of impurities contained in it (compare curves 8 and 4).

In contrast to contact fatigue, in the process of testing for low-cycle fatigue life, the best results were demonstrated by samples based on unalloyed ABC100.30 iron powder with a microaddition of Na (figure 2, b; curve 5). The lowest values of $N_{lcf}$ are observed on the reference samples. Similarly, to contact endurance, microalloying made it possible to increase the $N_{lcf}$ values (figure 2, b; compare curves 4 and 8).

When microalloying Ca and Al, the highest indices of contact and low-cycle fatigue life are observed for samples obtained from AstaloyCrM powder (figure 2, e, f; in figure 2, c, d, the data for $N_{90}$ and $N_{lcf}$ are not shown). The introduction of microadditives Ca and Al, as well as microadditives of Na, makes it possible to increase the values of $N_{90}$ and $N_{lcf}$ in comparison with the reference samples. The mechanism of the positive effect of microadditives on the characteristics of contact and low-cycle fatigue life is associated with the activation of cohesive interaction on interparticle surfaces [11]. The introduction of Na or Ca microadditives contributes to the refinement of grain boundaries and particles from impurities. In addition, the presence of Na or Ca microadditives makes it possible to activate the diffusion of carbon in powder steels [13]. This contributes to the formation of a more homogeneous structure, which is a significant factor in terms of ensuring the stability of the indicators of mechanical properties [9]. Aluminum, being localized on interparticle surfaces in the composition of a solid
solution in iron, provides an increase in free energy and activation of recrystallization processes that control the formation of cohesive bonds.

Figure 2. The effect of carbon content on rolling contact (a, c, e) and low-cycle (b, d, f) fatigue life of hot-deformed powder steels and their stability. Base powder: ABC100.30 (1, 5); AstaloyCrM (2, 6); PZhV2.160.26 (3, 4, 7, 8). \( C_{Na} = 0.2 \text{ wt. \%} \) (a, b); \( C_{Ca} = 0.3 \text{ wt. \%} \) (c, d); \( C_{Al} = 0.4 \text{ wt. \%} \) (e, f). \( N_{V} \) (1 – 3, 7); \( N_{f0}, N_{f0f} \) (4 – 6, 8). 7, 8 – samples-witnesses without microadditives of alloying elements

The effect of carbon and microalloying additives included in the composition of powder steels on the stability indicators of the characteristics of contact and low-cycle fatigue life described above is somewhat different. For steels with microadditives of Na or Ca, the dependences of the stability indicators of the values of contact fatigue on the carbon content have a non-monotonic character (figure 2, a, c; curves 1 - 3). With an increase in \( C_{Ca} \) to 0.8 wt. % the \( V \) values decrease, reaching a minimum. At \( C_{Ca} < 0.8 \text{ wt. \%} \) the structure of steels is martensite with troostite patches. Despite the presence of a surface cemented layer with a homogeneous martensitic structure and individual inclusions of cementite, deformation of the wear tracks occurs in the process of contact loading of such samples.
The inhomogeneity of the distribution of troostite and martensite sections in the sublayer of the samples predetermines the corresponding inhomogeneity of the deformation of the wear tracks. In this regard, for low-carbon steels, the V values are at a rather high level. The development of pitting cracks is characterized by branching with the formation of a large number of fracture products. At $C_{cm} = 0.8$ wt.%, the relief of the fracture surfaces decreases, which determines the presence of a corresponding minimum in the V ($C_{cm}$) dependences. Microalloying made it possible to increase the stability of the parameters of the contact fatigue of steels based on PZhV2.160.26 powder with a high content of impurities in comparison with the reference images (figure 2, a; compare curves 3 and 7).

The dependences of the stability indicators of the low-cycle fatigue life values of steels with microadditives of Na or Ca on the carbon content are monotonic (figure 2, b, d; curves 1 - 3). As $C_{cm}$ increases, the stability of the low-cycle fatigue life characteristics decreases (the V values increase). In this case, in the process of testing low-carbon steels, a fatigue crack originating in the surface layer near non-metallic inclusions, during its development, forms a fracture zone characterized by the presence of areas of shear transcryllalline and ductile fracture.

The presence of a viscous component in the fracture contributes to an increase in the stability of the $N_{lcf}$ values. The result obtained is consistent with the known concepts of the dependence of the stability of strength indicators on the absolute value of the latter: as strength increases, its stability decreases [14].

The effect of the Al microadditions on the stability indicators of the endurance characteristics of powder steels is ambiguous. In contrast to steels with microadditives of Na or Ca, the stability of the values of contact fatigue decreases monotonically with an increase in the carbon content: the values of V increase in the entire studied range of the $C_{cm}$ values (figure 2, e; curves 1 - 3). This is due to the fact that aluminum and carbon during mixing and subsequent cold pressing of the blanks are localized near the interparticle boundaries. In the process of sintering and hot forging, aluminum, being a graphitizing element, causes the formation of zones of structural inhomogeneity near the former interparticle boundaries depleted in carbon. The distribution of these zones in the structure of the samples does not have an ordered character, which predetermines the corresponding instability of the contact endurance indices, which increases with an increase in $C_{cm}$.

The impurity content is an additional factor that determines the level of stability of properties. Therefore, in all cases, their most stable indicators were obtained on samples made of ABC100.30 powder with a low content of impurities (curves 1). The least stability of properties was demonstrated by samples of PZhV2.160.26 powder with a high content of impurities (curves 3).

The dependences of the stability parameters of the low-cycle fatigue life characteristics on the carbon content are non-monotonic (figure 2, f; curves 1-3). As $C_{cm}$ increases in the range of 0-0.4 wt. % values of V fall, and with a further increase in $C_{cm}$ the V values increase. This is due to the fact that the $N_{lcf}$ characteristic is more determined by the quality of interparticle jointing compared to the contact endurance. Therefore, at $C_{cm} = 0.4$ wt. % the factors ensuring the activation of the processes of cohesive interaction dominant.

The mechanism of the positive effect of Al has been described above. The action of small additions of carbon can also have a positive effect in connection with the processes of reduction of oxides on interparticle surfaces [9]. The combined effect of these two factors (microadditives of Al at $C_{cm} \leq 0.4$ wt.%) provided an increase in the stability of the $N_{lcf}$ values. Obviously, in the process of testing for contact endurance, the action of this mechanism was leveled by the action of another mechanism described above and associated with the “pushing” of the solid surface cemented layer by balls of the test stand into the body of the softer sublayer and the core of the sample, as a result of which a large number of wear products were formed.

With a further increase in $C_{cm}$ above 0.4 wt. %, the stability of the $N_{lcf}$ values decreases, which is associated with an increase in the structural inhomogeneity near the interparticle boundaries.
4. Conclusion

1. The introduction of Na or Ca microadditives contributes to an increase in the stability and absolute value of the indicators of the contact and low-cycle fatigue life of HDPS due to the activation of cohesive interaction on interparticle surfaces and diffusion of carbon, which contributes to the formation of a homogeneous structure.

2. The presence of a viscous component in the fracture of samples of microalloyed low-carbon powder steels obtained as a result of testing for low-cycle fatigue provides a higher stability of the durability characteristics in comparison with high-carbon steels destroyed by the mechanism of transcrystalline cleavage. When testing for contact fatigue, a higher stability of the durability indicators is observed on high-carbon steels, which is associated with the specifics of the stress-strain state of the surface layers of the samples under contact loading.

3. The introduction of Al microadditives contributes to an increase in the contact and low-cycle fatigue life of HDPS in comparison with the reference samples. However, the formation of zones of structural heterogeneity in steels containing above 0.4 wt. % C, causes a decrease in the stability of these characteristics.

4. The optimal combination of the absolute value and stability of the contact and low-cycle fatigue life indicators is demonstrated by samples of eutectoid composition steels with Na or Ca microadditives, obtained on the basis of AstaloyCrM atomized chromium-molybdenum powder.

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