Research on the hot-deformed powder materials splicing surfaces’ quantitative characteristics

T V Goncharova*, Yu N Bandura, D A Pashyan, N G Prous and V V Sukhomlinova
Don State Technical University, 1, Gagarin sq., Rostov-on-Don, 344000, Russia

E-mail: ya_germes@mail.ru

Abstract. This article presents an experimental determination of the powder materials splicing surfaces’ quantitative characteristics obtained by hot processing.

This study analyzes the hot deformed powder material’s microstructures. We consider what changes the powder grains have undergone and what are the single crystals of different sizes, a certain configuration and color, as well as the crystallographic planes’ direction with respect to the thin section plane. In the course of the work, spheroidization of particles and their coagulation due to the absorption of small particles by large particles have been observed.

The influence of the initial particles’ size on the structure formation of powder alloys by using the homogenization features during sintering has also been investigated. The deformation character of the porous steel particles’ material compacts from two types of a mixture based on coarse and fine iron powder with different porosity is considered. As a result, after sintering, an increase in semi-finished products and a corresponding increase in porosity were observed. In this case, the effect nature of the dependence on the porosity of samples of powders of various sizes on plastic deformation is traced on thin sections of the porous semi-finished products. With a greater degree of compaction, the activation of the surfaces collapsing during hot stamping of pores and their rapid coalescence after reaching physical contact are detected.

The work revealed that the investigated non-porous hot-deformed steel is almost uniform in structure due to the complete deformation of all particles of various sizes original iron powder. The clear boundaries of the particles’ coalescence surfaces on the studied samples are traced.

In the course of this study, certain dependences of the splicing quality on interparticle contact surfaces during hot pressure treatment of powder semi-finished products and the properties of the obtained hot-deformed powder materials on the considered technological parameters [1, 2], for example, such as the particles’ size, determining the interparticle splicing surfaces boundaries length, differences in the mixture particles’ deformability, leading to a decrease in the volume of material deformed during hot processing pouring powder semi-finished products.

Introduction

Technological methods for the creation of powder materials such as dynamic hot pressing and transverse hot stamping, based on hot pressure treatment of porous semi-finished products, are especially promising and significant among the powder metallurgy methods [3]. The above-mentioned methods are based on a similar mechanism of fundamental phenomena responsible for the structure formation of a material: compaction, deformation and splicing on the interparticle contact surfaces.
The formation of the powders’ technological characteristics is greatly influenced by such a parameter as the particle size distribution of the powder, which relates to physical properties.

An important scientific task in the study of parameters affecting the structure formation is the study of a quantitative assessment of the criteria for the coalescence of hot-deformed powder materials and the possibility of their mutual correlation [4]. Other works have attempted to solve this scientific aspect. Such a question as the quantitative characteristics of splicing surfaces and the quality of interparticle contacts formed after hot pressure treatment of porous articles is a problem not fully studied. All of the above-mentioned led to the use of the materials obtained in additional scientific research and calculations [5].

Main part
The possibility of the splicing surfaces quantitative value experimental determination is based on a clearly traceable difference in the boundaries of these surfaces on thin sections of the hot-deformed and even for the material that has undergone the heat treatment after hot stamping, providing intergranular splicing and migration of the former grain boundaries.

An example of this situation can be shown as the samples of powdered perm-alloy 50N having the chemical composition shown in Table 1, in the state after hot stamping and additional annealing (1100°C, 3h) in the dissociated ammonia environment. A sprayed perm-alloy powder was used, corresponding distribution to the sprayed iron powder in grain size, with the particle size class 315 (Table 2).

Table 1. The chemical composition of perm-alloy 50N, mass %.

|    | Ni   | Si    | Mn    | Cu | C  | P   | S    | Fe   |
|----|------|-------|-------|----|----|-----|------|------|
| 49.0-50.5 | 0.15-0.30 | 0.3-0.6 | 0.2 | 0.03 | 0.02 | 0.02 | other |

From this powder without additional additives by the cold pressing at the pressure of 600 MPa, the semi-finished products with a porosity of 20% were formed to obtain the hot-deformed ring samples with the dimensions d=35×25 mm, h=5 mm, used later for magnetic measurements. The semi-finished products were heated before hot stamping at 1100°C for 7 min in dissociated ammonia.

An analysis of the hot-deformed powder material’s microstructures shows that the powder grains after hot stamping practically did not undergo any significant changes and are the single crystals of different sizes with a clearly defined morphology of a faceted configuration and different color, determined by the direction of the crystallographic planes with respect to the thin section plane. The content of the structural separations corresponds to the content of the particle size fractions in the powder. In separate large particles, twin phases are visible in the form of white plane-parallel inclusions.

Table 2. Granulometric composition of iron powder.

| A method of powder manufacturing | Size class | The output of the fraction, %, when the particle size, mm |
|--------------------------------|------------|--------------------------------------------------------|
| Reco-vered                      | 450        | 600-0.450 0.450-0.315 0.315-0.250 0.250-0.200 0.200-0.160 0.160-0.100 0.100-0.071 0.071-0.044 < 0.045 |
|                                | 160        | 0.045       |
| Sprayd                         | 450        | 0.600-0.450 0.450-0.315 0.315-0.250 0.250-0.200 0.200-0.160 0.160-0.100 0.100-0.071 0.071-0.044 < 0.045 |
|                                | 315        | 0.05       |
|                                | 200        | 0.02       |
After annealing in the samples’ structure, the particles’ spheroidization is observed, their coagulation due to the absorption of the small particles by the large ones. The boundaries of the migrated splicing surface are identified as the boundaries of new grains; previously existing boundaries are visible as a dotted dark rash along the former particle contours. It can be additionally noted that the boundaries of large particles migrate, and the small ones are absorbed by them. A noticeable spheroidization of small and large particles is also observed.

Quantitative dependence $l_s = f(\bar{l})$ for perm-alloy powder dispersed in fractions with an average particle size $\bar{l} = 300, 200, 120$ and $65 \, \mu m$, is shown in Fig. 1 (curve 1).

The dependence is described by an analytical equation of type (1) with the value $k_s \approx 7.5$, which confirms its reliability.

$$l_s = \frac{\sum l_i}{\sum S_i} = k_s \frac{\sum l_i}{\sum S_i} ,$$

where $l_i$ is the specific length of splice boundaries, $k_s^1$ defines the proportionality coefficient to determine $l_i$ in size $l_i$; $l_i$ is the $i$-particle size, $S_i$ is the single particle surface, $l_u$ denotes the particle perimeter.

![Figure 1](image)

**Figure 1.** Dependence $ic=f(l)$ for powders: 1 – permalloy; 2 – quick cutting steel R6M5K5; 3 – furnace charges became G13p.

Similar relationship $l_s = f(\bar{l})$ was obtained by processing high-speed steel R6M5K5 samples fractures’ fractograms. The semi-finished products’ sample were obtained by electric-discharge sintering [6, 7] of the fractions powders 1–8 (Table 3), and then they were subjected to hot stamping on a hammer with the values of the compaction work $290–310 \, MJ / m^3$, providing an almost non-porous state of the hot deformed powder material.

The samples fractures’ fractograms from the fractions’ powders 1 and 2, are the most characteristic, and the dependence $l_s = f(\bar{l})$ is shown in in Fig. 1 (curve 2). Their analysis shows that even hot deformation did not lead to a significant distortion of the particle shape, which led to almost complete coincidence of the experimental values $l_i$ with the calculated value obtained by the formula (1) at $k_s=4$. Since the spherical particles have the smallest possible perimeter of the splices surfaces boundaries in a section with a fixed area, the value $k_s$ for the same particles is the smallest, and the curve function (1) occupies the leftmost position in the coordinates $\bar{l} - l_s$.

**Table 3.** Fractional composition of powder P6M5R5 and the average fractions’ particle size

| Fractions No. | Particle size range, microns | The average particle size, [microns] | Content, mass [%] |
|---------------|-----------------------------|-------------------------------------|-------------------|
The possibility of studying the influence of the initial particles’ size on the structure formation of powder alloys from a mixture based on powder mixture was realized for steel G13p by using the features of its homogenization during sintering. Its temperature (1200 °C) exceeds the melting point of ferromanganese. For several seconds, molten ferromanganese moistens the surface of the iron powder particles, flowing into micropores, which leads to a reduction in the diffusion paths of manganese and provides the alloy homogenization during sintering for 10–40 minutes (depending on the iron particles’ size). Carbon diffusion occurs simultaneously. In addition, it should be borne in mind that in the process of sintering compacts of iron powder at temperatures above 600 °C recrystallization actively proceeds [8]; therefore, inside the particles between the grains disappear and only the boundaries between the individual particles remain. Similar processes occur during the high-manganese compacts’ sintering [9, 10]. Thus, sintered moldings of steel G13p particles are subjected to hot stamping, the sizes of which can be taken equal to the particle sizes of the initial powder. Dependence

\[ l_s = f(I) \]  

shown in Fig. 1 (curve 3), which can be described by the formula (1) with the value \( k_s \approx 6.6 \).

To study the material deformation nature of the porous compacts’ particles made of G13p steel, the samples \((d = 20 \text{ mm, mass } 25 \text{ g})\) of two types of charge based on coarse and fine iron powder with porosities of 31 and 26%, respectively, were prepared. After sintering, an increase in semi-finished products and a corresponding increase in porosity to 36 and 30% were observed. The sintered semi-finished products after heating for 5-7 minutes at 1200 °C subjected to dynamic compaction in the stamp. The magnitude of the given work is chosen so that the samples after compaction have different porosities: (25-50) MJ / m³ – (24-26) %; (100-120) MJ / m³ – (14-15); (250-300) MJ / m³ – (0.5-1.0) %.

In the samples’ structure, the deformed volumes of the particles’ material or their individual parts are clearly distinguished, which are identified as the corresponding sections of the thin section covered with characteristic shear lines. With high porosity of the samples from both coarse and fine powder, almost the entire volume of the compacts’ material almost did not undergo any plastic deformation: shear lines that are the traces of slip planes during deformation are not visible on thin sections. The deformation of the workpiece in this case occurs mainly as a result of filling the technological gap between it and the matrix walls, as well as due to structural deformation, in which the compression particles are displaced as a whole. But even with large porosity in some places of the workpieces, which are apparently characterized by a favorable orientation of the octahedral planes of particles (grains) with respect to the direction of the acting forces, the individual volumes’ deformation is observed.

With a greater degree of compaction, almost complete coverage by the particles’ volumes deformation can be traced, while it can be traced not so much in the contact zones as in the pore particles, capturing almost completely their volumes. It is this circumstance that causes the activation of the surfaces collapsing during hot stamping of pores and their almost instantaneous fusion after reaching physical contact. Such deformation of particles occurs due to the flowing of their material into the pores, and it is obvious that the flowing conditions are facilitated with increasing pore size. This is clearly seen on the structures and is in good agreement with the formula (2), according to which the pore size increases with the coarsening of particles at the same porosity of the compacts.

|   | 600   | 6.54  | 1.72  | 7.35  | 11.13 | 21.43 | 11.23 | 2.31  | 38.29 | 100.00 |
|---|------|------|------|------|------|------|------|------|------|--------|
| 1 | +450 |      |      |      |      |      |      |      |      |        |
| 2 | -450 | +355 |      |      |      |      |      |      |      |        |
| 3 | -355 | +250 |      |      |      |      |      |      |      |        |
| 4 | -250 | +160 |      |      |      |      |      |      |      |        |
| 5 | -160 | +70  |      |      |      |      |      |      |      |        |
| 6 | -71  | +63  |      |      |      |      |      |      |      |        |
| 7 | -63  | +56  |      |      |      |      |      |      |      |        |
| 8 | -56  |      |      |      |      |      |      |      |      |        |
| PA|      |      |      |      |      |      |      |      |      |        |
\[ V_p = V_u \frac{p}{1-p} \]

where \( V_p \) is the pore volume, \( V_u \) is the particle volume.

The structure of practically non-porous hot-deformed steel is characterized with full coverage of all particles volumes deformations, which indicates its uniformity inherent in both the particle size distributions of the initial iron powder. It is possible to trace the boundaries of the particles’ surfaces on the samples, i.e., the surfaces of adhesion. Along the way, it is necessary to note the experimental confirmation extreme scarcity of the deformation uniformity during hot stamping, which is mentioned in many publications.

**Summary**

The above-mentioned studies confirm the presence of a determining influence of technological parameters on the splicing quality, as well as the formation of the structure and properties of the powder materials obtained by hot pressure treatment. In the present work, the main attention was given to an in-depth study of the splicing surfaces’ quantitative characteristics. The analytical expressions were used to determine them during the experiments.

The recognition of the splice surfaces boundaries is fraught with some difficulties. In some cases, they coincide with the grain boundaries, in others they can be recognized solely by epy implicit indicators, especially when it comes to intracrystalline splicing and its surface displacement.

As a result of epy experiments on sintered powder semi-finished products on the effect of different sizes of powder particles on pore sizes, as well as some features of the near-pore volumes material’s deformation, the kinetics of deformation processes due to the transformation of particle and pore sizes was revealed, while the enlargement of such inevitably leads to a much larger volume deformation material.

**References**

[1] Dorofeev V Yu, Loginov V T, Goncharova T V, Chumakov V I 1996 Influence of technological factors on the quality of splicing of hot-deformed powder materials *Study of the problems of improving road transport, Novocherkassk, SRSPU* 35-37.

[2] Goncharova T V 2001 Features of the influence on the quality of the coalescence of technological conditions upon receipt of hot-deformed powder steels, Powder and composite materials. Structure, properties, technology, *Novocherkassk, SRSPU* 48-52.

[3] Dorofeev Yu G 1977 Dynamic Hot Pressing (Metallurgy, Moscow).

[4] Dorofeev V Yu, Loginov V T, Goncharova T V 1997 A quantitative assessment of the criteria for the fusion of powder materials and the possibility of their mutual correlation, the Use of new materials in mechanical engineering *Novocherkassk, SRSPU* 35-37.

[5] Goncharova T V 1996 Factors that determine the quality of hot-deformed powder materials, criteria for evaluating it and ways to improve it (Thesis ... cand.techn.sc, Novocherkassk, SRSPU).

[6] Dorofeev Yu G, Bogdanchenko A N 1984 Obtaining semi-finished products for hot stamping from hard-deformed powder materials *Powder metallurgy* 2 60-64.

[7] Dorofeev Yu G, Bogdanchenko A N 1985 Optimization of the density of billets obtained by electric-discharge sintering from sprayed hardly deformed powders *Powder metallurgy* 2 57-61.

[8] Fedorchenko I M, Pugina P I, Filatova N A, Yurchenko A G 1968he structure of metal-ceramic materials based on iron (Metallurgy, Moscow).

[9] Zherditsky N T 1981 Study of the formation of the structure of structural materials and products obtained by dynamic hot pressing (Thesis ... Doc.techn.sc, TsNIICHM, Moscow).

[10] Shorshorov M Kh, Krasulin Yu L, Dubasov A M et al. 1967 On the question of the estimated estimation of pressure welding modes *Welding production* 24-28.