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INFLUENCE OF HIGH-SPEED SINTERING ON THE STRUCTURE AND PROPERTIES OF POWDER STEELS

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A B S T R A C T

As the technology of sintering steels is carried out at different temperatures and conditions, the for-mation of their structure and properties are somewhat different. Proper sintering technology allows the pro-duction of high-density and high-strength powder steels. In most cases, the reason for the decrease in the pro-perties of powder steels is that the diffusion process, which occurs as a result of low sintering temperatures and low sintering times, is weak or non-existent. As we know, density and many physical and mechanical properties of the product increase as the pores close by dif-fusion during sintering. Proper welding technolo-gy allows the production of high-density and high-strength powder steels.

K E Y W O R D S

Sintering;
Technology;
Powder;
Steel; Structure;
Properties; Oil;
Gas; Industry.

1. Introduction

Many technologies are used to produce high-density molybdenum powder steels. The most modern of these technologies is Spark Plasma Sintering (SPS), which combines pressing, sintering and, in most cases, heat treatment modes. The main advantage of this method is the combination of several technological operations and a significant reducation of the time spent on the process. The properties formed during the sintering process by the SPS method are higher and more durable than other methods. As an advantage of the process, it is shown that there are op-portunities to make very complex shaped details and products by this method. The main purpose of the work is to study the possibility of obtaining high-density and properties of grinding steels using the SPS method [1,2].

Due to the low porosity of Astaloy grade grinding steels, these steels are widely used in the oil and gas industry in the manufacture of parts for various devices and equipment. Thus, the high density, as well as the low content of nickel and molybdenum, has proven itself in the oil and gas industry as a material with high corrosion resistance and high properties. Astaloy powder smelting steels in the oil and gas industry, heating exchange, color apparatus, employment apparatus, reactor equioment, pipe furnaces products, main oil pump, pump equipment, different applications, for highways, oil pipelines, etc. can be offered for the manufacture of certain parts of the device.

2. Research method

Spark plasma sintering technology (SPS) has been used in powder metallurgy for several years. With this sintering technology, it is possible to sinter grinding products consisting of submicrons and nanowires. Currently, there is a wide range of literature on SPS sintering technology. Loo-king at this literature, it is possible to observe the production of high-performance powder steels, both simple and complex properties [3]. In general, along with plasma sintering technology, FAST (Field Assisted Sintering Technology) sintering technology with electric heaters) technology is also widely used. The main advantages of FAST and SPS sintering technologies are the implementation of the heating process in a very short time, the possibility of heat treatment in a short time and the possibility of automatic adjustment of the production of small-powder steel structure [4]. The strength and toughness of powder steels sintered by these methods is much higher than that of conventional sintering. The pressmold made of graphite in the sintering process with SPS and FAST technology, which allows to obtain such details and products from solid alloys [5].

The experimental part. The FCT HP D 250/1 brand sintering device, which consists main-ly of electric heaters, is used for high-speed sintering of powder steels. The operating temperature of this device is 2500 °C and is equipped with a special vacuum chamber (fig. 1). Chromium powder steels, which are widely used in powder metallurgy and are characterized by high properties, were used for the experiments. The properties of steels sintered by this method (strength, toughness, crack resistance, etc.) are much higher than those of steels sintered by other methods. Chrome-plated steel with a diameter and height of 30 mm was used in the sintering device, and the sintering temperature was 1250-1300 °C. The total heating and storage time during the sintering process was 2.6 minutes [3,4]. Figure 1 gives an overview of the plasma sintering device's vacuum chamber and the working process. As can be seen in the figure 1, the device consists of a vacu-um chamber (1), a pressmold made of graphite (2), a lower punch (3) and an upper punch (4).

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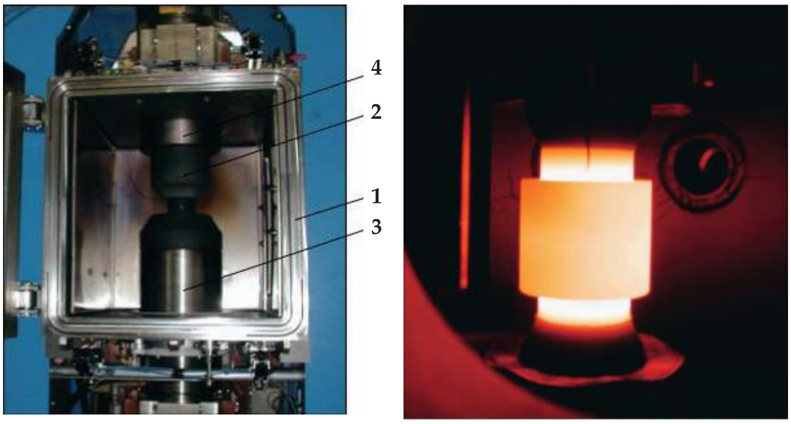


Fig. 1. View of the vacuum chamber of the Spark Plasma Sintering (SPS) device [4] 1 - vacuum chamber body, 2 - press-mold made of graphite, 3 - lower punch, 4 - upper punch

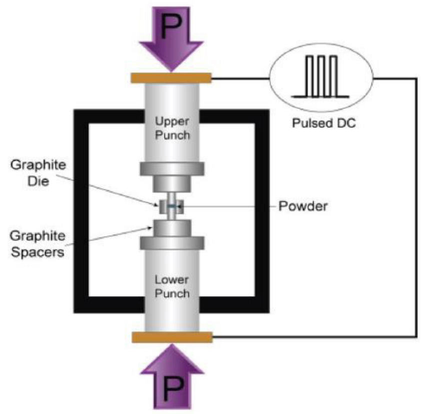


Fig. 2. Schematic diagram of the SPS apparatus [4]

Table

Spark plasma (SPS) sintering technology for powder steels ASTELOY (1.75% Ni, 1.5% Cu, 0.5%Mo, 0.7%C)

N	Argon pressure, Bar, 1 bar =10^5 Pa	Sintering temperature, °C	Sintering time, sec	Storage time, sec	Density of steel, gr/cm^3
1	0.1	1250	140	300	7.67
2	0.1	1250	140	300	7.69
3	0.1	1300	100	160	7.72
4	0.1	1300	120	60	7.75
5	0.1	1300	100	60	7.78

Figure 2 shows a schematic description of the SPS device. The device mainly consists of a vacuum chamber, lower and upper punches, graphite press mold.

Molybdenum powder steels often contain tungsten and chromium, and as a rule, the amount of molybdenum in such steels varies from 0.5 to 1.5%. When the amount of molybdenum in the composition of refined steels is more than 1.5%, the structure of the steels consists of ferrite, and molybdenum, together with iron, form a compound of Fe_3Mo_2 and FeMo intermetallics, which contain 53.2% Mo and 63.2% Mo, respectively [6]. Molybdenum in molybdenum powder steels increa-ses the concentration of carbon in the perlite and directs point S to the left in the Fe-C case diagram. Molybdenum is a strong carbideforming element and can easily combine with carbon to form MoC and Mo_2C carbide. Extraction of these carbides occurs mainly when the amount of molybdenum in steels is 8-10% [7]. However, when the amount of molybdenum exceeds the socalled amount, the formation of 3C binary carbides in cementite (Fe, Mo) intensifies. The amount of carbides in the structure of molybdenum grinding steels can be changed with the help of heat treatment regimes. At a sintering temperature of 500 °C, molybdenum steels first form Fe_3C carbide, and as the sintering ti-me increases, the formation of Mo_2C carbide also intensifies, which increases the dispersion of the carbides mainly as the sintering time increases. During sintering, molybdenum grinding steels have very little solubility of molybdenum in γ and α-iron, due to the fact that the area of α-iron is very high compared to the area of γ-iron. The intensity and diffusion of the solubility of carbon and mo-lybdenum in γ-iron occurs at a sintering temperature of 1000 °C. However, this diffusion also occurs at 1000-1200 °C, and the diffusion coefficient of molybdenum is further intensified during recrystallization of iron [3]. As the initial temperature of martensite conversion increases in all molybdenum steels, the diffusion coefficient of molybdenum increases significantly relative to carbon. However, this diffusion does not have a significant effect on the thermodynamics of the perlite structure. Although the fragility of molybdenum grinding steels increases, their strength, corrosion resistance and wear resistance increase. This is due to the fact that dispersed molybdenum carbides are formed in the structure and occupy the entire phase. The more dispersed the carbides, the finer the temperature of the quenching and annueling. However, when molybdenum is added to some molybdenum powder steels, the plasticity of the steels increases [4].

Chromium-molybdenum powder steels were sintered at 1300 °C in argon for 2 minutes. After sintering, the steels were quenched at 180 °C. Carbonyl iron powder with a size of 3 microns, C-1 grade colloidal graphite powder with a size of 7.6 microns and molybdenum powder with a size of 0.9 microns were used in the powder cast. In general, during sintering, the diffusion coefficient of carbon in γ-iron in this type of steel is higher than in molybdenum [4]. The presence of $Me_{23}C_6$ and Me_3C carbides in the structure of this type of steel leads to an increase in microhardness. During the sintering of a number of powder steels, its structure consists of sorbitollike perlite, and depending on the amount of molybdenum in the composition, the degree of dispersion of carbides in the structure is higher than that of chromium-molybdenum steels. This is due to the formation of secondary carbides in the structure, their distribution in the solid solution and the cost of the cooling rate [5].

Figure 3 shows the technology of sintering of refractory

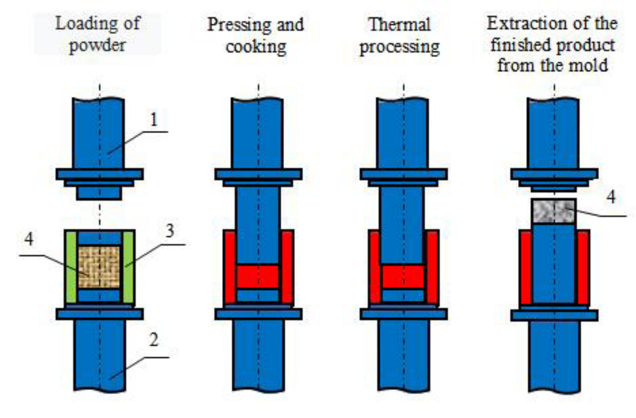


Fig. 3. SPS technology of smelting steels (plasma method) model of sintering 1 - top punch, 2 - bottom punch, 3 - special matrix, 4 - kneading, 5 - finished product

steels by SPS method. As can be seen in the figure, the pulp is pressed and sintering in a working chamber. Next, the heat treatment process is carried out. Then the final product is obtained. When the firing temperature is maintained at 500 °C, molybdenum steels first form Fe_3C carbide, and as the firing time increases, the formation of Mo_2C carbide also intensifies, which increases the dispersion of the carbides mainly as the firing time increases [6].

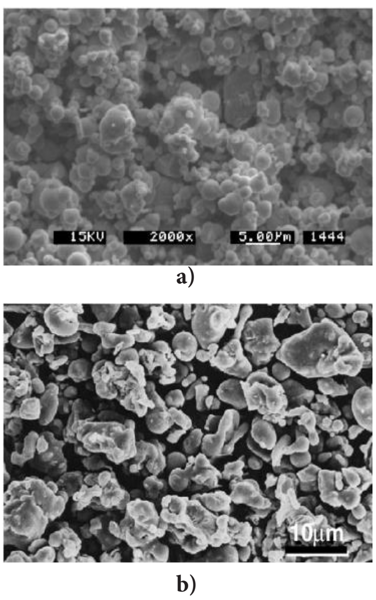
Figure 4 shows an SEM micrographs showing of ASTELOY powder steel with 1.75% nickel, 1.5% copper, 0.5% molybdenum and 0.7% carbon added. Improvement of one or more pro-perties often makes deterioration of other properties inevitable. Therefore, it is impossible to achi-eve the maximum value of all properties in the same steel. Therefore, it is necessary to correctly determine the optimal value of a group of properties that are most needed in accordance with the operating conditions and location, and, if possible, try not to let other properties fall too low.

The molten tool steel is dusted in an inert gas environment to prevent oxidation, resulting in rapid cooling of the liquid small grains and selfheating. The obtained high-dispersion steel is spherical in shape, varies in diameter from 10 to 600 microns and can contain up to 0.02% oxygen. The pulp is pressed in special containers (maximum diameter 500 mm and length 1700 mm) in a cold state under a pressure of 400 MPa. The container is then vacuumed, heated to 1150-1200 °C and repressed under a pressure of 140-150 MPa [7].

The size of carbides in welded and weakly deformed molybdenum grinding steels is very small (up to 2-3 microns) compared to conventional steels (8-20 microns) and is ideally distributed (less than 1 point). After all technological processes, the size and distribution of carbides in alloy steels do not change. In highly deformed steels, weak carbide lines are formed along the tensile direction. The improvement in the structure of the carbide phases is due to the fact that the amount of carbon and alloying elements in the metalbased martensite and residual austenite is much higher than in conventional steels. Therefore, the possibility of eutectic formation in steel is difficult, the amount is reduced. In addition, because the size of austenite grains is very small (1-2 microns), the perimeter of their boundaries increases, and the thickness of the eutectic reaches a minimum (1-1.5 microns) [6,7].

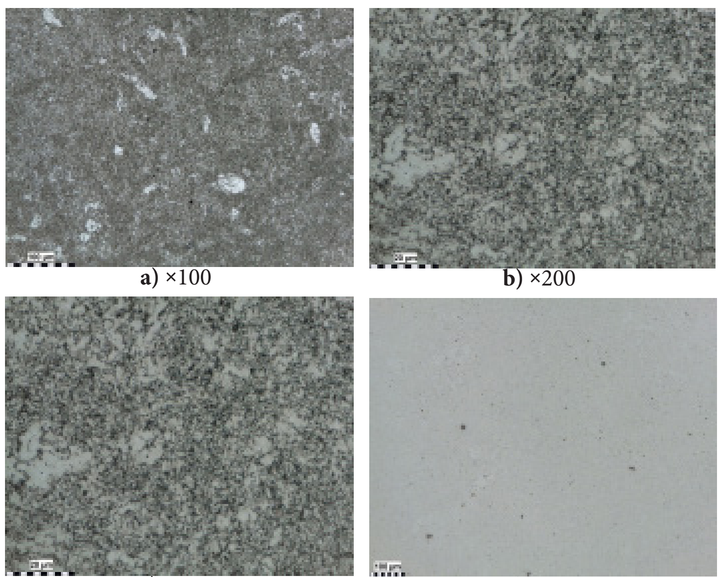
The polishing productivity of molybdenum steels made of rubble increases by 2-3 times, the purity class of the processed surface by 2 times and the corrosion resistance by up to 30%. This is explained by the increase in the surface area of solid carbide particles and thus the decrease in the risk of their disintegration in working languages. The secondary hardness and heat resistance of welded steels are almost indistinguishable from those of molten molybdenum steels, which are melted by conventional methods and are determined by their chemical composition. Therefore, the efficiency of smelting metallurgy is usually high for high-carbon and high-alloy steels with low mechanical and technological properties [8].

Metallographic studies have shown that the structure of chromium-molybdenum alloy steels consists of ferrite-perlite. At the same time, the value of pore porosity varies in the range of 0.5-15 (fig. 5). Relative porosity in grinding materials and products is determined by the ratio of the total volume of pores to the volume of the given piece of porous steel or products. Many researchers accept that the upper limit of



a) b)

Figure 4. SEM micrographs showing powder steels containing powder steels ASTELOY (1.75% Ni, 1.5% Cu, 0.5%Mo, 0.7%C)



a) ×100 **b)** ×200 **c)** ×1000 **d)** ×100

Fig. 5. Microstructure of containing powder steels ASTELOY (1.75% Ni, 1.5% Cu, 0.5%Mo, 0.7%C)

porosity in welded molybdenum steels is 0.5, and say that this limit does not affect the integrity and structural strength of the steel [6]. Therefore, steels with a porosity of more than 0.5 are called high, steels with a porosity of less than 0.1 are called low, and steels with a porosity of less than 0.01 are called microporous (cast) steels. All steels, including welded (non-porous) and compact (cast) steels, may have some micropores and non-metallic alloys of various origins. During hot and cold pressing, they sometimes become a source of new pores. Therefore, cast and welded steels are porous to one degree or another, and their mechanism of plastic deformation is the same to the extent of mini-mizing porosity.

A variety of operating conditions of machines and apparatus in the oil and gas industry necessitates a rational choice of materials. From this point of view, the studied materials have great prospects. Due this technology obtained by the low porosity and high density of powder steels can be used in the manufacture of parts for various installations and equipments in the oil and gas industry. High density of obtained materials, as well as low nickel and molybdenum have proven themselves in the oil and gas industry as a material with high corrosion resistance and high mechanical properties. In the oil and gas industry, Astaloy powder steels can be used in the production of heat exchangers, reactor equipment, main oil pumps, pumping equipment, junction parts of main oil pipelines.

Conclusion

1. The main advantages of FAST and SPS sintering technologies are the implementation of the heating process in a very short time, the possibility of heat treatment in a short time and the possibility of automatic adjustment of the production of small powder steel structure.

2. During SPS method, the strength and toughness of powder steels are much higher than conventional sintering. The press-mold made of graphite in the sintering process with SPS and FAST technology, which allows to obtain such details and products from solid alloys. Chrome-plated steel with a diameter and height of 30 mm was used in the sintering device, and the sintering temperature was 1250-13000C. The total heating and storage time during the sintering pro-cess was 2.6 minutes.

3. Powder steels containing 0.5%, 1.0% and 1.5% Mo were used for the sintering process. Steels with 0.5% molybdenum were sintered at 250-750 °C, steels with 1.0% molybdenum at 500-1400 °C, and steels with 1.5% molybdenum at 1250-1300 °C. The sintering time was 2.5, 2.0 minutes and 2.60 minutes, depending on the composition. The compression pressures of the samples were 300 MPa, 500 MPa and 550 MPa, respectively.

4. It was determined that the obtained sinter materials have application prospects in the purchase of details of oil and gas equipment.

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Влияние высокоскоростного спекания на структуру и свойства порошковой стали

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Реферат

Поскольку технология спекания сталей осуществляется при разных температурах и условиях, формирование их структуры и свойств происходит несколько иначе. Правильный выбор технологии спекания позволяет получать стали высокой плотности и повышенной прочности. В большинстве случаев причиной снижения свойств спеченных сталей является слабый или недиффузионный процесс, возникающий в результате низких температур и малой продолжительности спекания. Как известно, плотность и многие физико-механические свойства стали увеличиваются по мере закрытия пор в результате диффузии при спекания.

Ключевые слова: спекание; технология; порошок; сталь; структура; свойства; нефть; газ; промышленность.

Yüksək sürətli bişirmənin ovuntu poladlarının struktur və xassələrinə təsiri

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Xülasə

Poladların bişirilməsi texnologiyası müxtəlif temperatur və şəraitdə həyata keçirildiyi üçün onların strukturunun və xassələrinin formalaşması bir qədər fərqlidir. Düzgün bişirmə texnologiyası yüksək sıxlıqlı və yüksək möhkəmlikli ovuntu poladlarının istehsalına imkan verir. Əksər hallarda ovuntu poladlarının xassələrinin azalmasının səbəbi aşağı bişirmə temperaturu və aşağı bişirmə müddətləri nəticəsində baş verən diffuziya prosesinin zəif olması və ya olmamasıdır. Bildiyimiz kimi, bişirmə zamanı məsamələr diffüzya nəticəsində bağlandıqca poladın sıxlığı və bir çox fiziki-mexaniki xassələri artır.

Açar sözlər: bişirmə; texnologiya; ovuntu; polad; struktur; xassə; neft; qaz; sənaye.