RT&A, Special Issue No. 7 (83),
Volume 20, May 2025

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

Subhan Namazov[1], Shahin Mashayev[1], Taleh Taghiyev[1]

•

[1]Azerbaijan Technical University, Huseyn Javid Ave.-25, Az-1073, Baku
subhan.namazov@aztu.edu.az, shahin.mashayev@aztu.edu.az, taleh.taqiyev@aztu.edu.az

Abstract

Due to the fact that the technology of sintering powder steels is carried out at different tem-peratures and conditions, the formation of their structure and properties is obtained some what dif-ferently. Proper preparation of the sintering technology allows to obtain high density and properties of smooth steel. In most cases, the reason for the reduction of the properties of powder steels is that the diffusion process, which occurs due to the low sintering temperature and sintering time, is weak or not at all. As we know, the density and many physical and mechanical properties of the product increase due to pore diffusion during sintering.

Keywords: effect, high-speed sintering, powder, steel, drilling, sintering, technology, molybdenium powder steel.

I. Introduction

The advantage of the process is the availability of the possibility of making details and products of very complex shapes by this method. The main purpose of the work is to investigate the possibility of obtaining high density and properties of ovate steels using the SPS method [1]. Many technologies are used to obtain high density molybdenum scrub steels. The most modern of these technologies is plasma method (SPS) sintering, which includes pressing, sintering and, in most cases, thermal proce-ssing modes. The main advantage of this method is the combination of several technological opera-tions and a significant reduction in the time spent on the procession. In the process of sintering by the SPS method (Spark-Plasma-Sintering), the formed properties become higher and more durable than in other methods.

For several years now, they have been using the plasma method (SPS) big laser technology in powder metallurgy. With this sintering technology, it is possible to sintering powder products consisting of submicron and nano powder. Currently, very extensive literature can be found on the technology of sintering by the SPS method. Looking at these literature, it is possible to ac-company the production of high-quality powder steels of both simple shapes and complex confi-gurations. In general, in addition to plasma sintering technology, they also widely use FAST (Fi-eld Assisted Sintering Technology) sintering technology with electric heaters [2]. The main ad-vantages of FAST and SPS sintering technologies are the possibility of heating in a very short ti-me, the possibility of thermal processing in a short period of time and the possibility of automatic adjustment of obtaining a small-grain steel structure [3]. The strength and toughness of powder steels sintering by these methods is significantly higher than when sintering by conventional met-hods. Press-mold made of graphite in the process of sintering with SPS and FAST technology creates conditions for obtaining such details and products from solid alloys by this method [4].

The Spark Plasma Sintering (SPS) method is an effective technique for the compaction of pow-der materials. A main characteristic of this method is the direct heating of the pressing tool and-or the sample by pulsed direct electrical current with low voltage. This results in high heating ra-tes and allows for short treatment times in order to obtain highly compacted sinter bodies. The material transport (e.g. by diffusion) occurring during the sintering process can also be used for performing chemical reactions. Especially the conditions during the SPS process allow the use of the method also as an alternative synthesis route for intermetallic compounds, of which, some can be obtained only with difficulties by other techniques.

The purpose of the work the MPI for Chemical Physics of Solids was the first institute in whe-re an SPS setup had been installed. Two SPS apparatus are available, one of them being installed inside an Argon-filled glove box, which make it very useful for the compaction and/or synthesis of air/moisture sensitive samples. Both machines allow for external forces up to 50 kN, direct current up to 1500 A and a voltage limit of 25 V with typical pulse length of 2.5-3 ms.

II. Research Methodology

The brand sintering (FCT HP d 250/1) unit, which consists mainly of electric heaters, is used for high-speed sintering of powder steels. The working temperature of this unit is 2300-2500^{0}C and is equipped with a special vacuum chamber (Figure 1). For the implementation of the experiments, chromed steels were used, which are widely used in powder metallurgy and have high properties. The properties of steels sintering by this method (strength, hardness, resistance to crack formation, etc.) were obtained much higher than steels sintering by other methods. The diameter and height of 30 mm of chrome-plated cast steel was used in the sintering unit, and the sintering temperature was 1200-1300^{0}C. The sintering process was to heat the holding time was 2.4-2.6 minutes in total. Powder steels containing 0.5-1.0% and 1.5% Mo were used for the sinte-ring process. Steels containing 0.5-1% molybdenum were baked at 200-700^{0}C, steels containing 1.0% molybdenum at 500-1450^{0}C, steels containing 1-1.5% molybdenum at 1250-1300^{0}C. The sintering time was 3.0-3.5 minutes and 2.25 minutes, depending on the composition. The pressing pressure of the samples was 300 MPa, 500 MPa and 550 MPa, respectively.

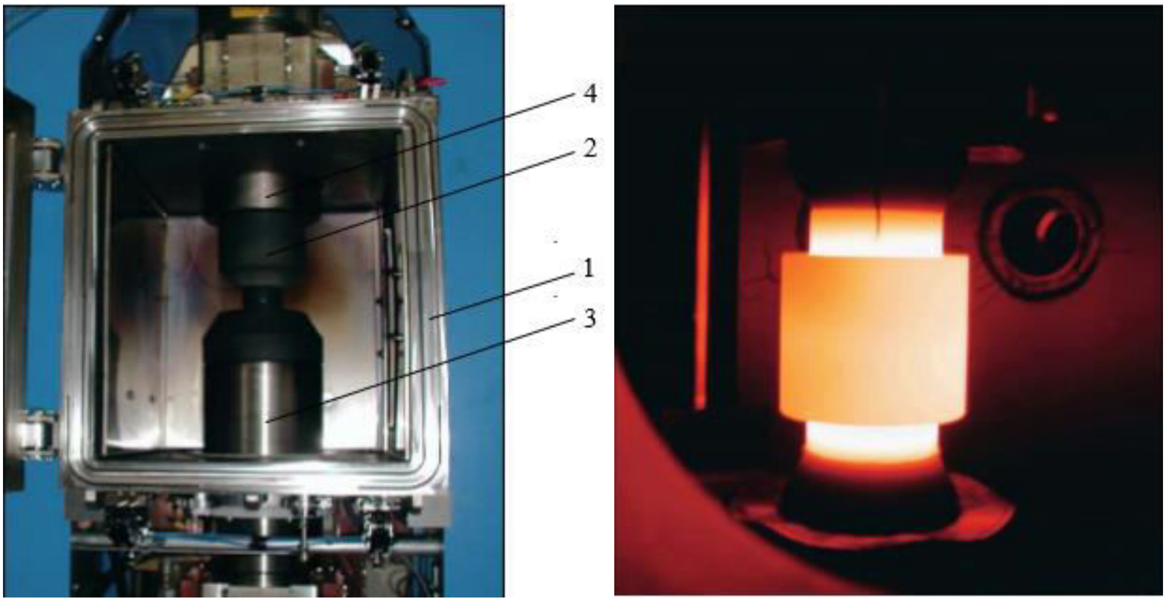
III. Discussion of the results obtained

Tungsten and chromium are often present in the composition of molybdenum scrub steels, and, as a rule, the amount of molybdenum in such steels varies in the amount of 0.5-1.5%. When the molybdenum content of powder steels is more than 1.5%, the structure of the steels consists of ferrite, and the molybdenum in the composition, together with iron, forms a compound Fe_3Mo_2 and FeMo intermetallide, which, respectively, contains 53.2% Mo and 63.2% Mo [5]. Molybde-num in molybdenum scrub steels increases the concentration of carbon in the compound in perlite and points the S point in the Fe-C case diagram to the left. Molybdenum is a strong carbidifying element and can easily combine with carbon to form MoC and Mo_2C carbide. The uptake of these carbides occurs mainly when the molybdenum content in steels is 8-10% [6]. However, when the amount of molybdenum is greater than the amount of deyelled, the formation of 3C dicycarbides in cementite (Fe, Mo) becomes even more intenvivilized. It is possible to change the amount of the named carbides in the structure of molybdenum scrub steels with the help of thermal proce-ssing modes.

When the sintering temperature is set at 500^{0}C, Fe_3C carbide is first formed in molybdenum steels, and as the sintering time increases, the formation of Mo_2C carbide is also intensified, which increases the dispersion of carbides mainly as the sintering time increases. In molybdenum scrub steels during sintering, the solubility of molybdenum in γ and α-iron is very low, and this is due to the fact that the α-iron rin area is extremely high in relation to the γ-iron area. The intensity and diffusion of carbon and molybdenum solubility in γ-iron occurs at a sintering temperature of 1000^0C. İs also found in cases where this diffusion occurs at 1000-1200^0C, and the diffusion coef-ficient of molybdenum intensifies even more during the recrystallization of iron [7]. As the initial temperature of martensite conversion increases in all molybdenum pofrets, the diffusion coeffici-ent of molybdenum rises significantly compared to that of carbon. However, this diffusion does not have a significant impact on the thermodynamics of the perlite structure. While the fragility of molybdenum scrub steels increases, their strength, corrosion resistance and inedible endurance increase. This is due to the fact that dispersed molybdenum carbides are formed in the structure, occupying the entire phase. The further dispersion of carbides, the increase in the temperature of tabulation and tabulation becomes even more crumbly per year. However, when molybdenum is added to some molybdenum scrub steels, the plasticity of steels rises [7]. The chrom-nickel-molyb-denum scrub steels of the brand П20ХН2М were baked at a temperature of 1250-1300^0C in argon mixture for 1.5-2 minutes.

Table 1: *By plasma method of powder steels containing 0,5-1,5% molybdenum sintering technology (SPS method)*

N	Argon pressure, Bar, 1bar =105 Pa	Sintering temperature, 0C	Sintering time, sec	Holding time, sec	Density of steel, qr/sm^3
1	0,1	1220	120	250	7,675
2	0,1	1230	125	270	7,694
3	0,1	1240	130	180	7,725
4	0,1	1350	135	65	7,757
5	0,1	1300	114	60	7,789



a) b)

Figure 1: *Vaccum of plasma sintering (SPS) device view of his camera (a)*
1-body of the vaccum chamber, 2-press-mold made of graphite, 3-lower score,
4-upper score. Sintering in a plasma method cooking (SPS) unit general view of the process (b)

After baking, the steels were subjected to steelmaking at 150-180^{0}C. Carbonyl iron powder with a size of 2.5-3 mkm, calloid graphite powder with a size of 7.6 mkm C-1 and molybdenum powder with a size of 0.75-0.9 mkm were used in the composition. In steels of this type during sintering in general, the coefficient of diffusion of carbon in γ-iron is higher than in molybdenum [8]. The presence of $Me_{23}C_6$ and Me_3C carbides in the structure of this type of steel leads to an increase in micromanagement. When baking a series of powder steel, its structure con-sists of perlite, similar to sorbite, and depending on the amount of molybdenum supplied to the composition, the degree of dispersibility of carbides in the structure is higher than that of pofrets with chrommolybdenum. The reason for this is the formation of the second carbides in the struc-ture, their distribution in the solid solution and the price of the cooling rate. This cooling tem-perature intensifies in the range of 400-500^{0}C, and as a result (Fe, Mo)$_2$C carbide is formed, resulting in the hardness of steel 1080-1230 HV, and the hardness of molybdenum carbide is 800-900 HV [9].

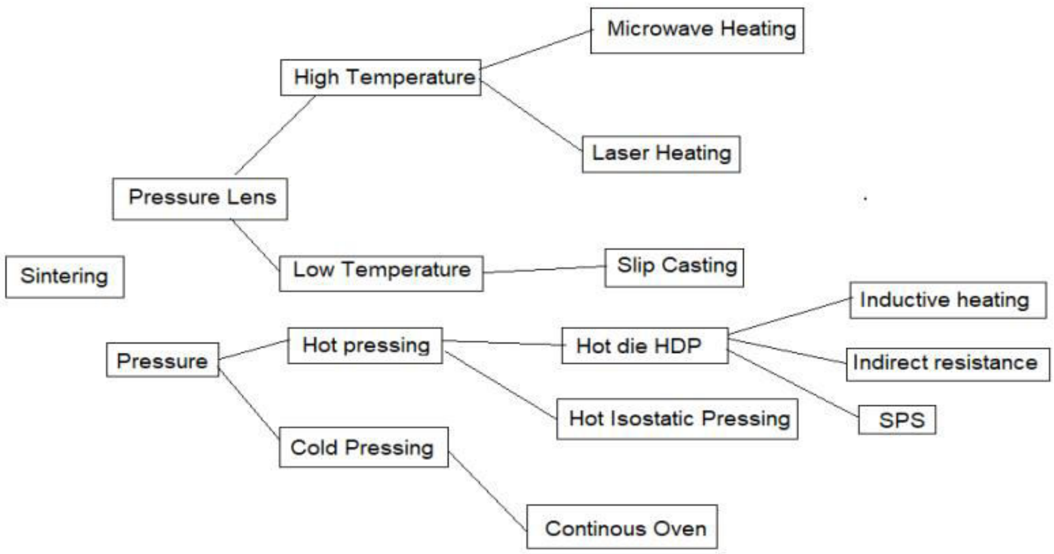


Figure 2: *Standard classification of sintered process*

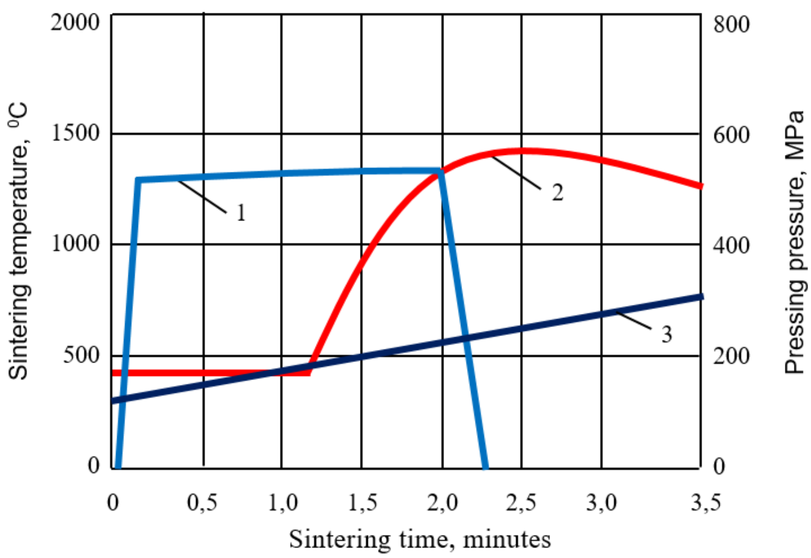


Figure 3: *Plasma method (SPS) sintering of powder steels containing different amounts of molybdenum 1-1,5% Mo, 2-1,0% Mo,3-0,5% Mo*

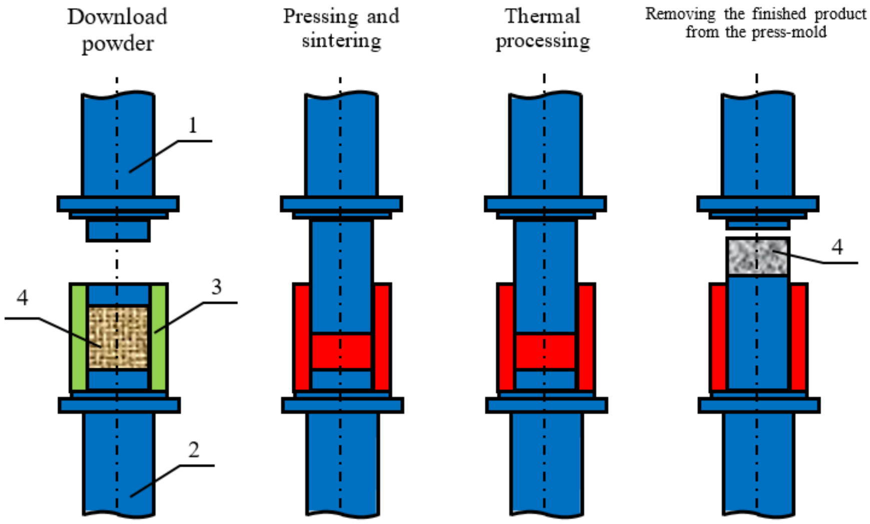


Figure 4: *With SPS technology of powder steels (by plasma method) model of sintering 1-top score, 2-bottom score, 3-special matrix, 4-powder glaze, 5-finished product*

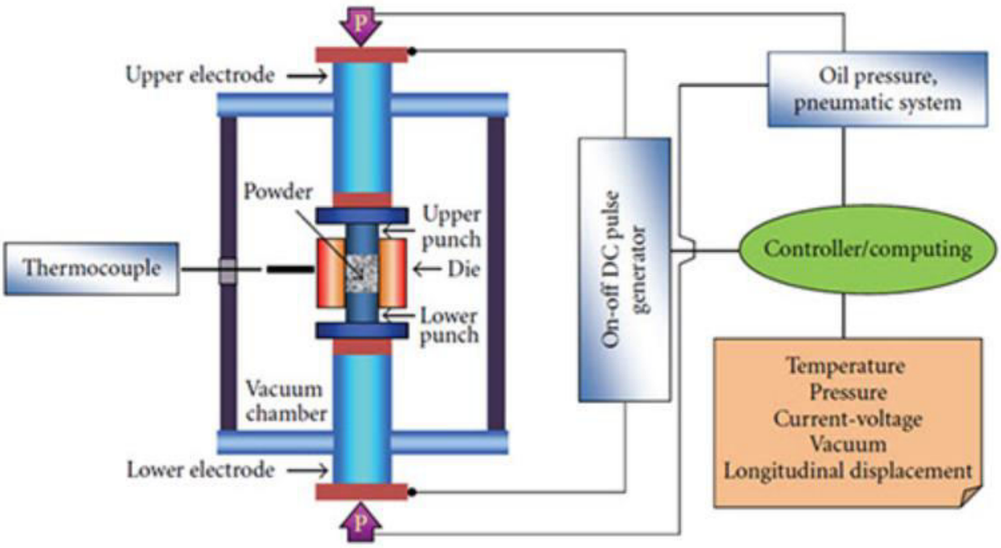


Figure 5: *Schematic of SPS process*

Control of sintering temperature is possible through setting the holding time, ramp rate, pulse duration, and pulse current and voltage. The DC pulse discharge could generate spark plasma, spark impact pressure, Joule heating, and an electrical field diffusion effect. In SPS, sintering is assisted by the on-off DC pulse voltage compared to conventional hot pressing as shown in Figure 5. The application of pressure helps plastic flow of the material. Figure 3 illustrates the flow of DC pulse current through the particles. Usually, SPS is carried out in four main stages. The first stage is performed to remove gases and create vacuum. Then pressure is applied in the second stage followed by resistance heating in the third stage and finally cooling in the fourth stage. When a spark discharge appears in a gap or at the contact point between the particles of a material, a local high-temperature state of several to ten thousands of degrees centigrade is generated mo-mentarily. This causes evaporation and melting on the surface of powder particles in the SPS pro-cess, and necks are formed around the area of contact between particles. The application of pres-sure and current, in addition to the high-localized temperatures generated through resistance pulse heating, improves heating rates and reduces sintering time and temperature leading to the console-dation of nanopowders without excessive grain growth. On the other hand, SPS is not only a bin-derless process, but also does not require a precompaction step. The powder is directly filled into a graphite die through which current is passed and pressure is applied leading to a fully dense material with superior mechanical properties [10].

IV. Conclusion

1. For the sintering process, powder steels containing 0.5-1.0% and 1.0-1.5% Mo are used. Steels containing 0.5% molybdenum were baked at 250-750^{0}C, steels containing 1.0% molyb-denum at 500-1400^{0}C, steels containing 1.5% molybdenum at 1250-1300^{0}C. The sintering time was 2.0-2.5 minutes and 2.60 minutes, depending on the composition. The pressing pressure of the samples was 300 MPa, 500 MPa and 550 MPa, respectively.
2. The main advantages of fast and SPS sintering technologies are the possibility of heating pro-cess in a very short time, the possibility of thermal processing in a short period of time and the possibility of automatic adjustment of obtaining a small-grain steel structure.
3. During plasma sintering (SPS method), the strength and toughness of powder steels is signify-cantly higher than when sintering by conventional methods. The press-mold made of graphite in the process of sintering using SPS and FAST technology creates conditions for obtaining such details and products from solid alloys by this method.
4. The diameter and height of 30 mm of chrome-plated cast steel was used in the sintering unit, and the sintering temperature was 1250-1300^{0}C. The heating and storage time during the sin-tering process was 2.0-2.6 minutes in total.

References

[1] Kessel, H.U., Hennicke, J., Schmidt, J., Weissgarber, T.,Kieback, B.F.,Herrmann, M., Rat-hel, J.: Feldaktiviertes Sintern „FAST"-ein neues Verfahren zur Herstellung metallischer und ke-ramischer Sinterwerkstoffe. Tagungsband 25. Pulvermetallurgisches Symposium, Hagen, 2006.

[2] J.,Kessel,H.U, Hennicke,.: Field Assisted Sintering Technology („FAST") for the Consolida-tion of Innovative Materials. cfi/Ber.DKG 81 (2004) [11] E14–E16

[3] Van-Meensel,K.,Kandukuri,S.Y.,Hennicke, J.,Anne,G., Jiang,D.,Vleugels, J.,Van der Biest, O.: Spark Plasma Sintering of Nanometer Size ZrO2-Al2O3-TiC0.5N0,5 Composites. EMRS 2004, September 6-10, 2004, Poland

[4] Nygren, M., Shen, Z.: Novel Assemblies via Spark Plasma Sintering. Silic. Ind. Spec. 69 (2004) 211–218

[5] Echeberria, J.,Martinez,V., Sanchez, J.M., Bourgeois, L., Barbier, G.,Hennic-ke, J. : Sintering Behaviour of Low Co Content cBNWC/ Co Composites by Either GE-HIP or FAST. Proc.of 16th International Plansee Seminar 2005, Eds. G.Knerin-ger,P.Rodhammer and H.Wildner, Vol.2, HM23, 434–448,(2005)

[6] Alvarez,M., Sanchez, J. M.: Densification of Nanocrystalline Ti(C,N) Powders with Nickel Aluminide Binder Phases Using Field Assisted Sintering (FAST). Submitted for publication to J.Amer. Ceram. Soc.

[7] L., Hennicke, Van Meensel, K.,Echeberria, J.,Sanchez, J.M., Martinez, V., Bourgeois, J., Kes-sel, H.-U.,Harden,P.,Van der Biest,O., Vleugels, J. : Field Assisted Sintering of Cubic Boron Nit-ride Dispersed Cemented Carbide (CDCC) Composites.EuroPM 2006.

[8] Dorofeev Yu.G. Dynamic hot pressing in cermet. Publishing house "Metalurgia",1986, 177 p.

[9] Zhornyak A.F., Radomyselsky I.D. Obtaining parts with high properties from powders. GOSINTI, M., 1964, pp.4-18.

[10] https://www.totalmateria.com/en-us/articles/spark-plasma-sintering/