

# A New Heat Treatment Cycle Design for High Wear Resistant in PM Steels

Onur Altuntaș1\*, Ahmet Güral<sup>2</sup>

0000-0002-4410-910X, 0000-0002-6211-8827

<sup>1</sup>Gazi University, Vocational School of Technical Sciences, Department of Machine and Metal Technologies, Ankara, Turkey <sup>2</sup>Gazi University, Faculty of Technology, Department of Metallurgical and Materials Engineering, Ankara, Turkey

#### Abstract

In this study, 1.5% by weight of natural graphite powders were added to Atomet 1001 pure iron powders and unalloyed high carbon powder metallurgy steel specimens were obtained. After the mixture was uniaxial pressed at room temperature and 700 Mpa pressing pressure, it was sintered in Ar protected atmosphere controlled oven at 1150 ° C for 20 minutes. The densities of the specimens before and after sintering were measured using precision scales and electronic calipers and presented graphically. After sintering, microstructure specimens containing primary cementite and typical dense perlite colonies were produced. The sintered specimens having primary cementite plus lamellar pearlitic structures were fully quenched from 950 °C temperature and then over-tempered at 705 °C temperature for 60 minutes to produce spherical-fine cementite particles in the ferritic matrix. After by this treatment, these specimens annealed at 735 °C temperature for 3 minutes were austempered at 300 °C salt bath for a period of 1 to 5 hours. After the heat treatment, dry sliding wear tests of specimens were carried out under 10N constant load and 500-1500m wear distance. At the end of the dry sliding wear test, the weight losses of the specimens were measured and it was determined that the dry sliding wear resistance increased with increasing austempering time after spheroidization annealing.

Keywords: Austempering, Microstructure, Powder metallurgy steel, Spheroidization

\* Corresponding author

Onur Altuntaş

onuraltuntas@gazi.edu.tr

Adress: Gazi University, Vocational School of Technical Sciences, Department of Machine and Metal Technologies, Ankara, Turkey

Tel:+903123548401

Fax: +903123543835

Research Article

 Manuscript

 Received
 13.04.2020

 Revised
 27.08.2020

 Accepted
 07.09.2020

Doi: 10.30939/ijastech..719815

## 1. Introduction

Alloy steels, commonly known as plain carbon steels, have vital proposals for a wide range of industrial applications, including automotive parts. The reason for this is lower costs than the alloyed steels. For some applications, however, their use is limited due to their low mechanical strength [1]. Porosity is commonly seen in materials produced by conventional powder metallurgy. However, it is well known that the pores adversely affect the mechanical properties because they cause a notch effect. porosity is one of the important parameters that directly affect mechanical properties. To reporosity in powder metal steels, changing the pressduce ing technique such as warm-hot pressing and bidirectional pressing, changing the sintering parameters such as increasing the sintering temperature, extending the sintering time, applying different thermal processes and increasing the pressing pressure [2-8].

Numerous properties of PM materials especially such as ductility, tensile strength, impact and fatigue are directly related to their pores, alloy elements and microstructure. These properties are obtained by applying various hardening mechanisms and proper thermomechanical procedures. [9-10].

A number of studies have been carried out on the effects of surface structure on wear and friction resistance of metallic materials in slippery contacts. The pores in powder metallurgy (PM) materials act as lubricants and increase abrasion together with friction resistance [11-12]. Many low alloy PM steels cannot be used as sintered. For this reason, the heat treatments are applied after the sintering to strengthen the PM steels. It is necessary to produce hard phases in a microstructure to increase wear resistance. To do this, the most common heat treatment applied to PM carbon steels is quenching + tempering [13-16].

Excessive hardness and brittleness of martensite formed by quenching process are removed by tempering. At the end

Altuntas and Güral / International Journal of Automotive Science and Technology 4 (3): 193-197, 2020 OVD AUTOMOTIVE ENGINE

of the heat treatment, the microstructure contains spherical precipitated cementite in the ferritic matrix. In steels with initial microstructure of primary ferrite-perlite or primary cementite-perlite, it takes a long time to spheroidize the cementite. However, the precipitation of spherical cementite particles is achieved by over-tempering the steels having the initial microstructure of martensitic [17-18]. A unique microstructure is formed between the austenite perlite and martensite transformation temperatures from eutectoid transformation temperatures. This structure, which is very different from perlite and martensite, is called "bainite" [19]. By definition, bainite is a very tough and ductile structure consisting of a mixture of ferrite and non-lamellar cementite (Fe<sub>3</sub>C). [20]. Bainite is a structure formed by the conversion of austenite at isothermal temperature under the perlite nose according to isothermal conversion diagrams. Cooling is diffusion controlled during austenite decomposition and perlite formation is an impossible. It also prevents the formation of martensite because cooling is sufficiently slow. [21]. Austempering is an alternative heat treatment to conventional quenching tempering heat treatments, especially to increase ductility and impact resistance despite certain levels of hardness and to reduce cracks occurring during quenching.

Hardness can be controlled depending on temperature and duration of tempering [17-19]. In general, the mechanical properties of a particular material are closely related to the microstructure and heat treatment process. Research on the effects on the mechanical properties of microstructures or heat treatment parameters has a significant engineering value [22-24].

It is intended that the steel materials having the microstructures of the invention have high hardness and strength due to the high toughness resistance of the bainitic microstructure as well as the spheroidized hard cementite phases. They are expected to have high abrasion resistance with hardness and increased strength. It is intended to be used as bearing material due to its superior abrasion resistance. In addition, due to both high toughness and hardness, it is aimed to spread to a wide range of applications, especially gun barrel materials in defense industry.

#### 2. Experimental Studies

For the experimental study, 1.5% by weight graphite and 0.5% Zn stearate as lubricant were added to Atomet 1001 pure iron powders produced by Sinter Metal company by water atomization method. The prepared powders were shaped to be abrasion test specimen according to ASTM G99 standard by uniaxial pressing at room temperature at 700 MPa pressure, and specimens having average density of 6.7 g.cm<sup>-3</sup> were produced and these specimens were produced in Argon gas atmosphere controlled oven at 1150 ° C for 20 minutes.

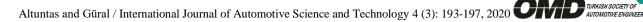
The high carbon (1.5% C) powder metal steel starting sample in the sintered state was first kept in the full austenite

zone at 950 ° C for 3 minutes, thus converting the crystallographic structure to the surface-centered cubic crystal austenite. It was then rapidly quenched at room temperature and converted into a volume centered tetragonal crystal lattice martensite. The martensite sample was annealed in an atmosphere controlled oven at 705 ° C for 1 hour to obtain spherical cementite in ferritic matrix and coded with M-705. In steels with initial microstructure of primary ferrite-perlite or primary cementite-perlite, it takes a long time to spheroidize the cementite. However, the precipitation of spherical cementite particles is achieved by over-tempering the steels with the initial microstructure of martensite. This method was used since spherical cementites were produced in ferritic matrix in a shorter time compared to conventional spheroidization heat treatment methods. The sample with this microstructure is partially austenitized at 735 ° C for 3 min after the  $\gamma$  + Fe<sub>3</sub>C phases on the Ac1 eutectoid transformation line in the Fe - C phase diagram and followed by rapid quenching at 300 °C neutral salt bath for 1-2 and 5 hours were coded as SCBM-1 and SCBM-2, SCBM-5 respectively.

Conventional metallography procedures were performed to reveal the microstructures, the specimens were etched with 3% Nital solution and the obtained microstructure imaging was determined by JEOL JSM-6060LV brand Scanning Electron Microscope (SEM).

After the sanding, polishing and etching processes, the hardness measurement of the phases and structures formed in the microstructure was done by HMV, SHIMADZU hardness tester. It was determined in vickers (HV1) by using 1 kg load as macro hardness. Arithmetic means were calculated by making at least 10 measurements for macro hardness measurements from each sample.

Dry sliding wear test specimens produced and prepared in accordance with ASTM G99-05 were subjected to abrasive abrasion testing by pin-on-disc. TRIBOMETER T10 / 20 was used for dry sliding wear tests. Hardox 500 steel discs with an average hardness of 55 HRc were used as abrasive counter surfaces. Before the abrasion test, the sample weights and the weight losses of the specimens due to the abrasion were made in SARTORIUS brand with a sensitivity of 0.0001 g. Dry sliding wear tests were carried out under constant 10N load, 500-1500m wear distance and constant 2.5 m / s sliding speed.



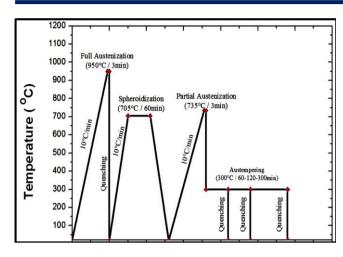


Fig. 1. Schematic heat treatment cycle graphics of SCBM specimens

## 3. Experimental Results and Discussion

Powder metal specimens with a density of 7.1 g.cm<sup>-3</sup> were produced. As in the literature [25], 92% theoretical density was obtained after sintering.

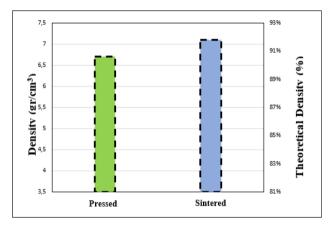


Fig. 2. Graphic of density variation of specimens

Figure 3a shows the microstructure of the sample formed by uniaxial pressing at room temperature and as in other studies [26-27], it was sintered at  $1150 \degree$  C temperature in Ar atmosphere. As seen from the microstructure, the expected primary cementite and perlite coverslips are seen in the sample after sintering. In addition, it is seen that pores, which are one of the most important problems of powder metallurgy, are present in the structure, albeit in a small number. In Figure 3b, martensitic specimens were produced by fully austenitizing at 950 ° C for 3 minutes followed by quenching. Due to the characteristic structure of martensite, high carbon content is formed as a sharp-edged plate in microstructure. Figure 3c depicts the full bainitic microstructure of a feathered structure that isothermally held in a neutral salt bath at 300 ° C by

standing in austenite zone. Figure 3d shows the microstructure of the M-705 sample where the martensite sample is spheronized in an Ar-protected atmosphere oven for 60 minutes. After 60 minutes of spheroidization heat treatment, spherical cementites were deposited in the ferritic matrix.

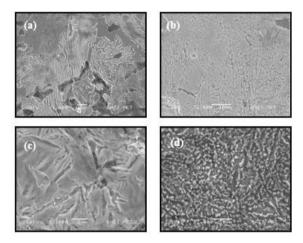


Fig. 3. SEM microstructures of (a) Sintered, (b) Martensitic, (c) Full Bainite (d) M-705 specimens

Figure 4a-c shows the microstructures of SCBM-1, SCBM-2 and SCBM-5. The aim of this microstructure design is to provide high toughness and strength due to the high toughness strength of the bainitic microstructure as well as spheroidized hard cementite phases. They are expected to have high dry sliding wear resistance with hardness and increased strength. As stated in the experimental studies, the production of spherical cementites in the bainitic matrix with different heat treatment cycles was realized. In all SCBM microstructures, the cementite particle size is about 1 um. With increasing austempering time, spherical cementites appear to become larger and more pronounced. As a result of 5 hours of heat treatment, it can be said that bainitic areas are more clear in microstructure.

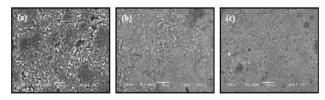


Fig. 4. SEM microstructures of (a) SCBM-1, (b) SCBM-2 and (c) SCBM-5 specimens

Table 1. Macrohardness values of s specimens (HV1)

Sintered	Martensite	Full	M-705 -1	SCBM		
		Bainite		1	2	5
$255 \pm 8$	530 ± 22	$450 \pm 14$	$225 \pm 9$	$312 \pm 21$	377 ±17	$445 \pm 11$

Altuntas and Güral / International Journal of Automotive Science and Technology 4 (3): 193-197, 2020



Table 2. Weight loss values of specimens due to friction
distance (g*10 <sup>-4</sup> )

	Sintered	Full Bainite	M-705 -1	SCBM		
				1	2	5
500M	20	7	10	3	3	2
1000M	36	12	17	7	5	4
1500M	39	20	33	11	10	10

The hardness and abrasion test results of the specimens are given in Table 1-2. The hardness of the sintered sample, which is the starting sample as expected here and contains dense perlite colonies, is 255 HV1 and is too low. The sample with the highest hardness was determined as 530 HV1 in the unstable balanced martensitic sample with very high dislocation density by giving instant water from the full austenite region as expected. However, the hardness of the M-705-1 sample with spherical cementite microstructure in the ferritic matrix obtained by annealing the martensite sample at 705 ° C for 1 hour was even lower than the sintered sample. This condition is thought to be caused by the hard lamellar hard primary cementites in the initial microstructure. The hardness of the sample with full bainitic microstructure was lower than the hardness of the martensite sample but 450 HV1 was determined as expected. Hardness of SCBM specimens increases with increasing austempering time after spheroidization. At the end of 1 hour austempering, it increased from 312 HV1 levels to 445 HV1 levels after 5 hours austempering. As can be seen from the microstructures (Figure 4a-c), it is seen that the volume ratios of spherical cementites deposited in the bainitic matrix have decreased with increasing austempering time. It is believed that soft spherical cementites dissolve in the bainitic matrix structure to reach thermodynamic equilibrium conditions and thus increased bainitic areas increase the rigidity of the structure. It is seen that the weight loss amounts of the specimens as a result of dry environment wear test are generally related with the hardness of the structure. In other words, wear resistance increases with increasing hardness. However, although SCBM specimens do not have the highest hardness, they show the highest abrasion resistance. It is thought that soft and ductile spherical cementites increase the wear resistance of the bainitic structure.

### 4. Conclusions

Spherical cementite particles in the bainitic matrix in high carbon steel processed by powder metallurgy. According to the experimental results obtained, the density of the sample sintered at 1150 ° C in Ar controlled oven and 7.1 g / cm<sup>3</sup> was determined to reach 92% theoretical density. The highest hardness value was determined as 530 HV1 in martensite sample depending on the heat treatments applied. In dry sliding wear tests, the minimum weight loss was observed in SCBM-5 sample due to increased

austempering time after spheroidization. It is considered that by increasing the isothermal annealing time and / or temperature further, the bainitic transformations will be increased and the mechanical properties of such high carbon powder metal steels will be significantly improved.

#### Acknowledgment

This study has been supported by the Scientific Research Project Program of Gazi University (under Project Number 65/2019-05). The authors are grateful to Gazi University for their financial support and the provision of laboratory facilities.

### References

- F. Ashrafizadeh, (2003). Influence of plasma and gas nitriding on fatigue resistance of plain Carbon (Ck45) steel, *Surface Coatings Technology* 174, 1196–1200.
- [2] Sarıtaş, S., Türker, M, Durlu, N., (2007). Powder metallurgy and particulate materials Processing, *Turkish Powder Metallurgy Association*, 2nd ed., Ankara, Turkey, 5-33, 217-230.
- [3] M.A. Erden B. Ayvacı, (2019). The effect on mechanical properties of pressing technique in pm steels, *Acta Polonica A*, 135(5): 1078-1080.
- [4] Uyumaz, A. (2015). An experimental investigation into combustion and performance characteristics of an HCCI gasoline engine fueled with n-heptane, isopropanol and n-butanol fuel blends at different inlet air temperatures. *Energy Conversion and Management*, 98, 199-207.
- [5] M. Türkmen, M.A. Erden, H. Karabulut, S. Gündüz, (2019). The effects of heat treatment on the microstructure and mechanical properties of Nb-V microalloyed powder metallurgy steels, *Acta Polonica A*, 135(4): 834-836.
- [6] D. Özdemir, S. Gündüz, M. A. Erden, (2017). Influence of NbC addition on the sintering behavior of medium carbon PM steels, *Metals*, 7 (4):121-132.
- [7] M.A. Erden, (2017). The effect of the sintering temperature and addition of niobium and vanadium on the microstructure and mechanical properties of microalloyed pm steels, *Metals*, 7: 329-345.
- [8] Erden M. A., (2017). Effect of pressing pressure on microstructure and mechanical properties of non-alloyed steels produced by powder metallurgy method. *Omer Halisdemir University Journal of Engineering Sciences*, 6(1), 257-264.
- [9] Gündüz, S.; Erden, M.A.; Karabulut, H.; Türkmen, M. (2016). The effect of vanadium and titanium on mechanical properties of microalloyed PM steel. *Powder Metallurgy and Metal Ceramics*. 55, 277–287.
- [10] S. Gündüz, H. Karabulut, M. Türkmen, (2016). Effect of the addition niobium and aluminum on the microstructure and mechanical properties of the micro-alloyed pm steels, *Materials* and *Technology*, 50(5): 641–648.
- [11]Pettersson U, Jacobson S. (2003). Influence of surface texture

Altuntas and Güral / International Journal of Automotive Science and Technology 4 (3): 193-197, 2020



on boundary lubricated sliding contacts. *Tribology International*; 36(11):857–64.

- [12]Xinmin Li, Ulf Olofsson.(2017) A study on friction and wear reduction due to porosity in powder metallurgic gear materials. *Tribology International*; 110, 86–95.
- [13]S. Tekeli, A. Güral. (2007). Dry sliding wear behavior of heat treated iron based powder metallurgy steels with 0.3% Graphite + 2% Ni additions. *Materials and Design* 28 1923– 1927.
- [14]M. Türkmen, M.A. Erden, H. Karabulut, S. Gündüz,(2019). The effects of heat trearment on the microstructure and mechanical properties of Nb-V microalloyed powder metallugy steels, *Acta Polonica A*, 135(4): 834-836.
- [15] M. Erdogan and S. Tekeli, (2002). The effect of martensite particle size on tensile fracture of surface-carburised AISI 8620 steel with dual phase core microstructure, *Materials and Design*, 23, 7,597.
- [16] M. A. Erden, S. Gündüz, H. Karabulut, M. Türkmen (2017).
   "Wear behavior of sintered steels obtained using powder metallurgy method. *Mechanika*. 23(4): 574-580.
- [17] Altuntas, O.. Güral, A. (2017).Effect of spheroidizing heat treatment on the microstructure, hardness and toughness of high carbon powder metallurgy steel. *Kovove Materialy*. 55, 5, 303-310.
- [18] Altuntaş, O. (2013). Investigation of spherinization heat treatment effects on microstructre and impact toughness properties on high carbon powder metal steels. Master Thesis, *Gazi Uni*versity Institute of Science and Technology.
- [19] Altuntaş, O., & Güral, A. (2019). Designing spherical cementite in bainitic matrix (SCBM) microstructures in high carbon powder metal steels to improve dry sliding wear resistance. *Materials Letters*, 249, 185-188.
- [20] Krauss, G. (2005). Steels: Processing, Structure, and Performance, ASM International.
- [21] Caballero, F.G.,Bhadeshia, H.K.D.H., Mawella, K.J.A., Jones, D.G., Brown, P. (2002). Very strong low temperature bainite, *Materials Science and Technology*, 18(3):279-284.
- [22] Sawa M, Rigney DA.(1987). Sliding behavior of dual-phase steels in vacuum and air. *Wear*, 119:369–90.
- [23] Türkmen, M., Erden, M.A., Karabulut, H. and Gündüz, S., (2017). Effect of TiN addition on the microstructure and mechanical properties of PM steels. *Technological Applied Sciences*. 12(4):178-184.
- [24] H. Demir, S. Gündüz, M. A. Erden, (2018). Influence of the heat treatment on the microstructure and machinability of AISI H13 hot work tool steel, *The International Journal of Advanced Manufacturing Technology*, 95 (5-8) : 2951-2958.
- [25]Erden, M. A., & Aydin, F. (2020). The investigation of wear and mechanical properties of carburized AISI 8620 steel by powder metallurgy. *International Journal of Minerals, Metallurgy and Materials.*
- [26] S. Gündüz, M.A. Erden, H. Karabulut and M. Türkmen, (2016). Effect of vanadium addition on the microstructure and mechanical properties of low carbon micro-alloyed powder metallurgy steels, *Materials Testing.*, 55, 4, 433.

[27] Erden M. A., Gündüz S., Türkmen M., Karabulut H. (2014). Microstructural characterization and mechanical properties of microalloyed powder metallurgy steels, *Materials Science and Engineering A*, 616: 201-206.