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

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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

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 reduce porosity in powder metal steels, changing the pressing 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
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 perlitic and martensite transformation temperatures from eutectoid transformation temperatures. This structure, which is very different from perlitic and martensitic, is called "bainite" [19]. By definition, bainite is a very tough and ductile structure consisting of a mixture of ferrite and non-lamellar cementite (Fe₃C). [20]. Bainite is a structure formed by the conversion of austenite at isothermal temperature under the perlitic nose according to isothermal conversion diagrams. Cooling is diffusion controlled during austenite decomposition and perlitic 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⁻³ 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 γ + Fe₃C phases on the A₃ 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.
3. Experimental Results and Discussion

Powder metal specimens with a density of 7.1 g.cm\(^{-3}\) were produced. As in the literature [25], 92% theoretical density was obtained after sintering.

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 °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 spheroidized in an Ar-protected atmosphere oven for 60 minutes. After 60 minutes of spheroidization heat treatment, spherical cementites were deposited in the ferritic matrix.

Figure 4a-c shows the microstructures of SCBM-1, SCBM-2 and SCBM-5. The aim of this microstructure design is to produce 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.

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

Table 1. Macrohardness values of s specimens (HV1)
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/cm³ 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.

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