EFFECT OF HEAT TREATMENT ON THE STRUCTURE AND MECHANICAL PROPERTIES OF PM Fe-Si-B COMPACTS THROUGH VACUUM CARBURISED

K. Widanka, J. Hejna

Abstract

This paper was aimed at determining the influence of heat treatment on structure and selected mechanical properties of sintered steel obtained through vacuum carburising of iron compacts with additions of boron and silicon.

Vacuum carburising with immediate sintering of the compacts made of a mixture of iron ASC100.29, ferroboron and silicon powders was carried out at 1050 and 1150°C in a laboratory vacuum furnace. The effect of quenching in oil and low-temperature tempering on the structure of surface layer and core, as well as selected mechanical properties of the sintered steel was studied. As a result of applied heat treatment, an increase of 74% of ultimate tensile strength, from 712 MPa to 1228 MPa, was obtained.

Keywords: vacuum carburising, iron-boron-silicon compacts, microstructure, mechanical properties, heat treatment

INTRODUCTION

A secondary heat treatment often is used to optimize the physical and mechanical properties of the ferrous powder metallurgy components after sintering. The most popular secondary heat treatments, commonly applied to PM parts, include hardening by austenitizing and quenching, processes that enhance surface properties (case hardening methods of carbonitriding, carburising, and nitriding), tempering, steam treating, annealing and brazing. Successful post-sintering heat treatment of PM parts involves the proper selection of material, heat treating parameters and heat processing equipment.

There are many factors that influence the heat treatment of PM parts and that ultimately determine the properties that can be obtained. Among them the most important are density and microstructure, which is the result of chemical composition and sintering process parameters. In particular the density of PM parts (and exactly porosity), which is related to its thermal conductivity, plays an important role when evaluating the response of material to heat treatment. High-density PM parts (with low porosity) have a high thermal conductivity, which ensures fast heating and cooling. Components having low densities (with higher porosity) will take longer to heat and will dissipate their stored thermal energy more slowly. Consequently, hardenability will be negatively influenced as density decreases.

Hardening by austenitizing and quenching is used to increase strength and overall wear and abrasion resistance. Ferrous PM parts typically are oil quenched from austenitizing temperature.

K. Widanka, J. Hejna Wroclaw University of Technology, Institute of Materials Science and Applied Mechanics, Materials Science Division, Wroclaw, Poland

The vacuum carburising method ensures a faster carburising course, mainly thanks to higher temperature and lower hydrocarbon gas pressure during the process [1-5]. Carbon diffusion rate in iron and iron alloys is decided by its diffusion coefficient, which for carburising processes carried out at high temperatures (above 1000°C) is more than twice as great as for traditional gas carburising [6]. An addition of silicon in these alloys increases the carbon diffusion coefficient. All available data [7-9] confirm that a boron addition increases carbon activity in the Fe-C-B alloys. This allows for the assumption that an additive of boron increases the carbon diffusion coefficient like silicon. It is well known that both of these elements also increase the hardenability of steels.

In this paper, effect of quenching in oil from a fixed austenitizing temperature on structure and selected mechanical properties of through-carburised Fe-Si-B compacts is presented. Compacts with about 0.01% of boron and 1.0% of silicon were pressed to a lowest density of 7.2 g/cm^3 in order to minimize the interconnected porosity.

Carburising of the compacts carried out in a vacuum furnace was accompanied by the sintering process. Sintered steels were obtained this way, with variable carbon content on their cross-sections. Subsequently, these steels were oil quenched and low-tempered. Such obtained sinters were subjected to structural examination of the surface layer and core, hardness measurements and tensile tests to determine their basic mechanical properties.

MATERIAL AND EXPERIMENTAL PROCEDURE

Standard flat specimens for mechanical testing with shape and dimensions: 5.85 x 6.05 mm (in gage section) according to ISO 2740 were prepared from a mixture of iron powder ASC100.29 (Höganäs AB), ferroboron FeB16 powder and silicon powder Si AX0.5 (H.C. Starck) with average particle size equal to 3.5 μm. Double-sided compacting under pressure within 750 to 800 MPa was applied to obtain compacts with the lowest density of 7.2 g/cm^3. The set of eight specimens was performed. Their chemical composition and green density, which were measured by means of Archimedes method, are given in Table 1.

The vacuum carburising process was carried out in a laboratory vacuum furnace (made by Seco/Warwick). Parameters of the process were based on the results of the our own research, to obtain surface carbon content within the range from 0.7% to 0.8%. The selected parameters are given in Table 2.

The carburising atmosphere consisted of propane diluted with nitrogen. Working pressure in the furnace chamber was 2 kPa. Stable working pressure in the chamber during carburising was maintained by cyclic dosing the gas with constant flow rate of 110 dm^3/h. Cooling rate in nitrogen was 7°C/s. A diagram of the vacuum carburising process is shown in Fig.1.

The heat treatment of specimens, consisting of quenching in oil and low-temperature tempering, was performed in a typical laboratory electrical chamber furnace. The heating of specimens at austenitizing temperature was run in the protective argon atmosphere. The heat treatment parameters are given in Table 3.

Tab.1. Chemical composition and density of compacts.

Chemical composition			green density
wt % Si	wt % B	wt % Fe	[g/cm3]
1.0	0.010	remainder	7.25

Tab.2. Parameters of vacuum carburising.

Carburising temperature [°C]	Carburising time [min]	Diffusion temperature [°C]	Diffusion time [min]	Total carburising time [min]
1050	60	1150	120	180

Tab.3. Heat treatment parameters of specimens.

Quenching			Tempering		
Temperature [°C]	Time [min]	Cooling medium	Temperature [°C]	Time [min]	Cooling medium
850	30	oil	150	60	air

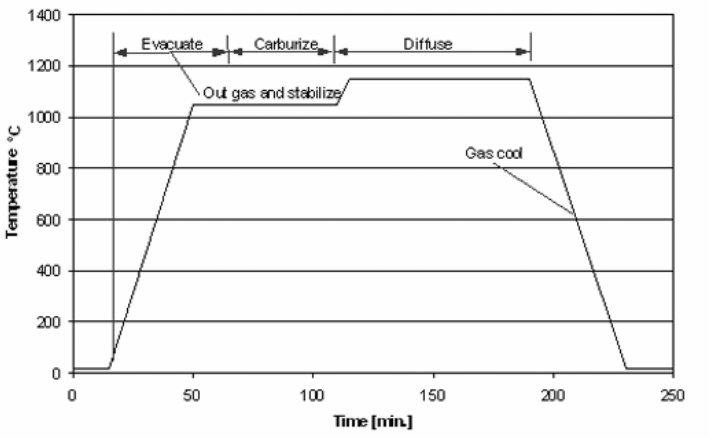

Fig.1. Schematic diagram of vacuum carburising process.

The specimens for structural examinations were taken from the specimens for mechanical tests after carburising (one series) and heat treatment (second series), by cutting fragments of their measurement parts. Metallographic cross-sections were made on transverse cross-sections of the specimens.

Microscopic examination of the specimens was performed using a light microscope Neophot 32 (by Zeiss) at magnification within 100 to 500 times and the SEM (Jeol 6610A) at magnification within 1000 to 5000 times.

Quantitative parameters of the microstructure were evaluated using a computerized image-analysis system "Multiscan" made by the Polish company Computer Scanning Systems. For each specimen a minimum of five images of microstructure were analysed.

Hardness measurements were taken using a Zwick hardness tester by the Vickers method at the load from 9.81 to 49.05 N.

Tensile tests were performed on a hydraulic pulsator MTS 810.

RESULTS AND DISCUSSION

The density of the carburised samples was determined by means of Archimedes method using a gas multipycnometer (Quantachrome Instruments). The measured density was 7.31 g/cm^3. Figure 2 shows a representative view of pores in the specimens.

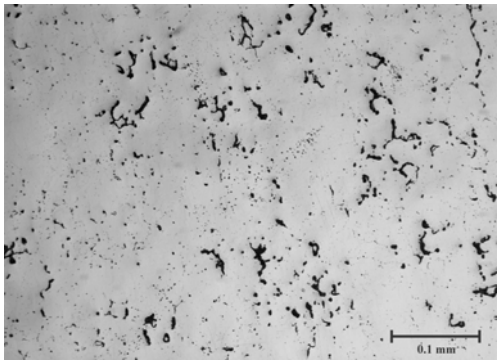

Fig.2. Representative picture of pores in the specimens.

Hardness was measured in two zones: in surface layer and in core. To determine the hardness profile, measurements were also taken on the cross-section along the centre line from the surface to the core every 0.25 mm. The obtained hardness measurements (in carburised state) permitted determining the thickness of the carburised layer. It was assumed that carburisation depth corresponds to mean hardness (HV 5) calculated as an arithmetical average of the values for the surface layer and core. The so-calculated thickness of the carburised layer is approx. 2.5 mm.

Hardness values for surface layers and cores of the specimens before and after heat treatment are given in Table 4, and representative hardness profiles HV 1 for the specimens are shown in Figs.3 and 4.

Tab.4. Hardness of the specimens before and after heat treatment.

After vacuum carburising		After heat treatment	
Hardness		Hardness	
of carburised layer HV5	of core HV5	of carburised layer HV5	of core HV5
195	138	595	412

After vacuum carburising the microstructure of surface layer of the specimens consists of pearlite and a small amount of cementite, probably of $Fe_3(CB)$ [10], precipitated on prior austenite grain boundaries during cooling (Fig.5). Core microstructure of specimens consists of ferrite with pearlite (Fig.6). Estimated fraction of pearlite and corresponding carbon content in the core of the specimens is 45% and 0.36%, respectively.

The carbon concentration in the core was calculated, accepting 0.77% C in the eutectoidal point and omitting silicon effect which shifts it to the left (according data in literature [12]), towards lower carbon concentration. With this effect considered, the calculated carbon concentration would be slightly lower.

The grains in the layer are larger than those in the core. The pearlite grain size evaluated by comparative method corresponds to the reference standard No. 7 in the layer and in the core to the standard No. 8, according to ASTM.

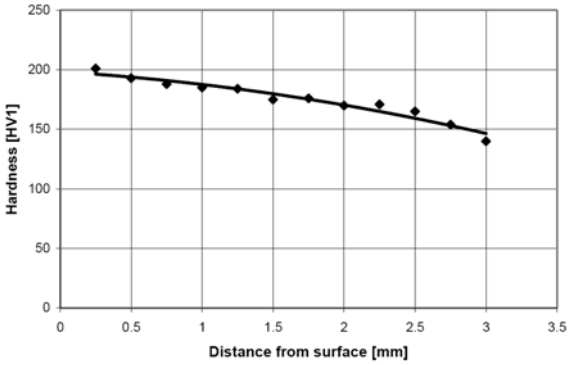

Fig.3. Representative hardness profile for specimens through vacuum carburised.

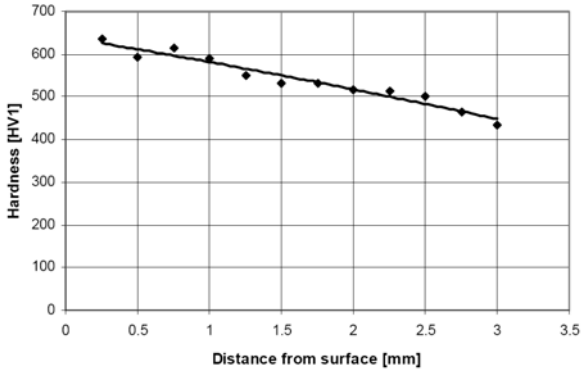

Fig.4. Representative hardness profile for specimens after heat treatment.

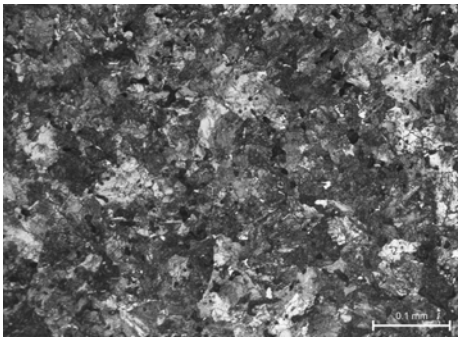

Fig.5. Carburised layer microstructure of the specimen. Nital etched.

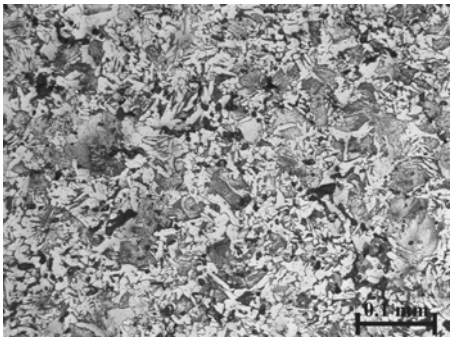

Fig.6. Core microstructure of the specimen. Nital etched.

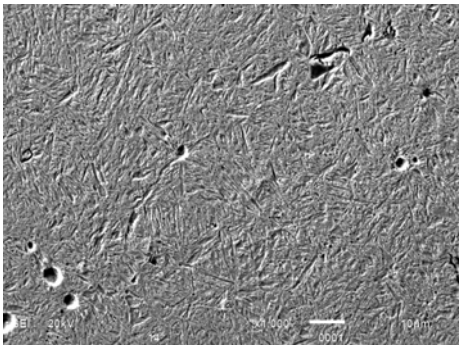

Fig.7. Carburised layer microstructure of the specimen after heat treatment. Nital etched.

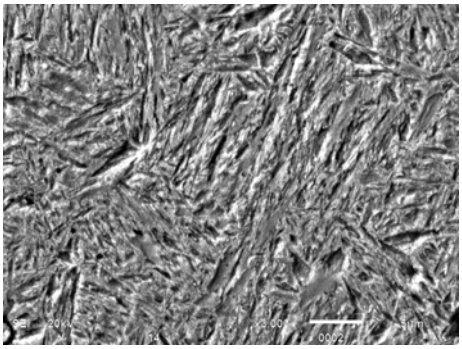

Fig.8. Carburised layer microstructure of the specimen after heat treatment (the same as in Fig.7. at higher magnification). Nital etched.

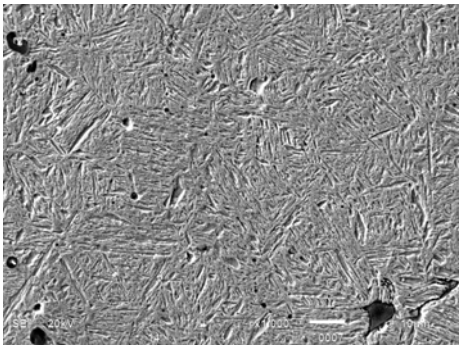

Fig.9. Core microstructure of the specimen after heat treatment. Nital etched.

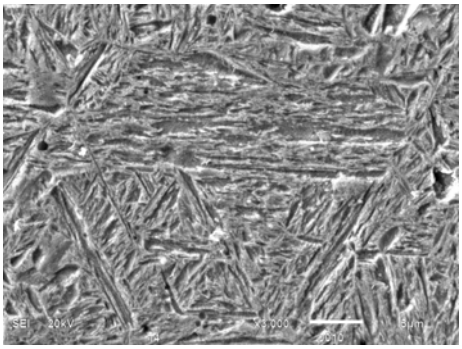

Fig.10. Core microstructure of the specimen after heat treatment (the same as in Fig.9. at higher magnification). Nital etched.

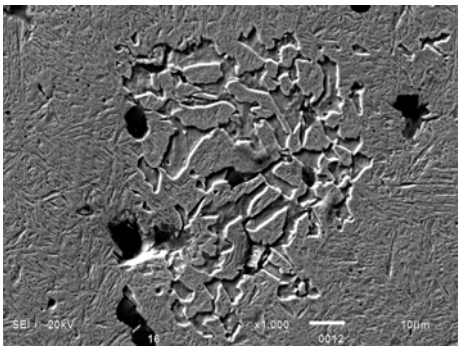

Fig.11. Core microstructure of the specimen after heat treatment. Ferrite islands. Nital etched.

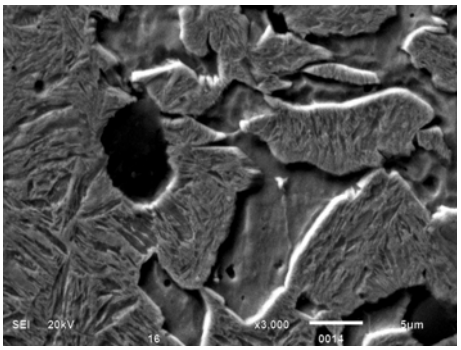

Fig.12. Core microstructure of the specimen after heat treatment. Ferrite islands (the same as in Fig.11 at higher magnification). Nital etched.

After heat treatment consisting of quenching in oil and low-temperature tempering, a microstructure of mixed martensite in the layer, as well as in the core was

observed. In the microstructure of the layer dominates the plate martensite (Figs.7, 8) and inversely in the core, where the lath martensite fraction is larger (see Figs.9, 10). In the core also, besides martensite, some ferrite islands are visible (Figs.11 and 12).

The presence of ferrite indicates that the selected austenitizing temperature was too low for a core, in which there is about 0.36% of carbon. This temperature of 850°C was read from Fe-Fe_3C diagram [11] omitting an effect of both boron and silicon additives. It is known (according to data in literature e.g. [12,13]), that these alloying elements increase the A_{c3} temperature (shift the G-S line up) in steels and therefore the austenitizing temperature should be higher than 850°C.

Basic mechanical properties of the specimens after vacuum carburising and heat treatment, denoted respectively with VC-S and HT-S, were determined in a static tensile test. The results are presented in Table 5.

Tab.5. Results of tensile test of the examined specimens.

Specimen	UTS* [MPa]	YS* [MPa]	Elongation* [%]
VC-S	712	431	3.6
HT-S	1228	Not determined	0.5

* Average values for three test specimens

As a result of applied heat treatment there was obtained an increase of 74% of ultimate tensile strength, from 712 MPa to 1228 MPa. This strength improvement is a consequence of the martensitic structure observed in the surface layer and core of the specimens. This structure is also responsible for a drop in plasticity measured by elongation. Both boron and silicon dissolved in prior austenite increased tempered martensite strength and hardness, and likely at the same time its brittleness, of which a symptom is the lack of yield strength and very small elongation. A beneficial effect to the hardness of tempered martensite by addition up to 1% silicon in wrought steels was found in [14]. The presence of ferrite islands in core microstructure decreases the material strength. This undesirable "weak" microstructure constituent is a potential place for microcrack nucleation during relatively light loads.

When comparing the obtained results with the properties of sintered steels produced by the traditional method (of mixtures of iron and graphite powders) based on the commercially available iron powders of similar density, it can be seen that they correspond to steels made of Distaloy powders (of grades SE and AE) with carbon concentration of 0.5 (constant on the entire cross-section). These steels include such alloying additives as copper (1.5%), nickel (4%) and molybdenum (0.5%). According to the database CASIP 5.1 (by Höganäs), tensile strength of these steels after similar heat treatment ranges from 1200 MPa to 1250 MPa, yield stress from 1120 MPa to 1150 MPa and elongation from 1.5% to 1.7%. Their hardness ranges from 430 to 450 HV. In comparison with these steels, it can be seen that the examined steel is more brittle after heat treatment. First of all, this is the result of different chemical composition as well as the more brittle tempered martensite. It seems, that the obtained strength of the examined steel might be even higher than 1228 MPa, if the core microsrtructure will be free of ferrite islands.

CONCLUSIONS

Application of the vacuum carburising method to iron compacts permits a combining of the carburising and sintering operations in one process. By proper selection of

the process parameters, carbon was introduced to the whole volume of the iron compacts containing additions of boron and silicon.

Such obtained specimens of sintered steels with variable carbon content on their cross-section, and thus with variable structure, were subjected to heat treatment to improve their strength parameters.

As a result of the applied heat treatment an increase of 74% of ultimate tensile strength was obtained. The achieved tensile strength of 1228 MPa is likely decreased because of ferrite islands present in the core microstructure.

These ferrite islands are the result of an incorrect austenitizing temperature, which was wrongly selected for the core of examined steel. On account of carbon content, the applied temperature of 850°C is proper for surface layer, and a bit too low for the core of steel, especially if the effect of silicon and boron on an increase of A_{c3} temperature will be considered. In order to eliminate ferrite, the steel should be austenitized from a higher than applied temperature, most probably within the range of 870°C to 900°C.

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