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Study of Different Vacuum Heat Treatments on the Strength of a Low Alloyed Sintered Steel

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Abstract. The present paper deals with the evaluation of different vacuum heat treatments on the microstructure of a low alloyed sintered Fe-[1.5 Cr-0.2 Mo]-0.6 C steel. Different gas pressures were applied on the material during cooling from the sintering temperature. The average cooling rates were calculated in the range of 1453 K to 673 K and were varying from 0.1 to 6 K/s, according to the gas pressure applied (0 to 600 kPa). Mechanical properties have been evaluated in terms of toughness, plasticity and hardness. The results show that increasing the nitrogen pressure resulted in an increased amount of bainite/martensite microstructure with high level of properties achieved.

Keywords. Powder metallurgy, vacuum heat treatment, microstructure, martensite.

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1 Introduction

Powder metallurgy (PM) has long been recognised as an effective technology for manufacturing parts to net or net shape parts. Some recent developments have extended the range of part shape which can be made by PM, along with processes that have extended the performance capabilities of PM parts. Alternative technologies are currently placing considerable emphasis on improving their energy efficiency, material utilization and dimensional precision [1–12].

Under certain circumstances the need for secondary hardening processes may be eliminated if the PM parts are cooled rapidly enough directly after sintering. The rate of cooling experienced by parts following sintering influences the microstructural constituents, which are formed. The microstructural constituents depend on the effective cooling rate, mass and shape of the part and material composition (in terms of hardenability) [13].

One of the most prospective methods is the vacuum heat treatment, especially in terms of low alloyed sintered steels using sinter hardening methods [14–16].

In addition to proper sintering atmospheres, some alloying elements, such as Cr, already applied with success in wrought steels, may be used for the improvement of mechanical strenght of PM materials.

Chromium have not been utilized to a large extent in the past due to their high affinity to oxygen, making it difficult to prevent oxidation during sintering, especially at lower sintering temperatures [6, 7, 9, 10, 15–18]. Hence, low oxygen partial pressures are required in order to reduce oxides on the powder surfaces and prevent further oxidation of the materials during sintering [6, 7, 9].

In any case, heat treatments are necessary to tune the mechanical properties to the final requirements and consequently, the effect of the heat treatments on their microstructure and mechanical properties has constituted a very dynamic research field during the last years. In this context, considerable efforts have been focused on the microstructural changes occurring during tempering, mainly in the martensite evolution.

The main aim of the present paper is to study the effect of different applied gas pressures (as an average cooling rate) after the vacuum sintering of a Fe-[1.5 Cr-0.2 Mo]-0.6 C steel.

2 Experimental Materials and Methods

Powder mixtures were homogenised in a Turbula mixer using Astaloy CrL powder (Höganäs AB), graphite powder and commercial AW wax powder as lubricant, then the final composition was Fe-[1.5 Cr-0.2 Mo]-0.6 C.

Specimens with a green density of approximately $6.56 \times 10^3\ \mathrm{kg \cdot m^{-3}}$ were obtained using a 2000 kN hydraulic press, applying a pressure of 500 MPa. Two different specimen types were prepared: "dog-bone" tensile (ISO 2740) and unnotched impact energy $55 \times 10 \times 10 \times 10^{-3}\ \mathrm{m^3}$ (ISO 5754). Specimens were debinded before sintering in Nabertherm type furnace.

In vacuum furnaces, the cooling rate is generally determined by the pressure of the gas (N_2) introduced into the

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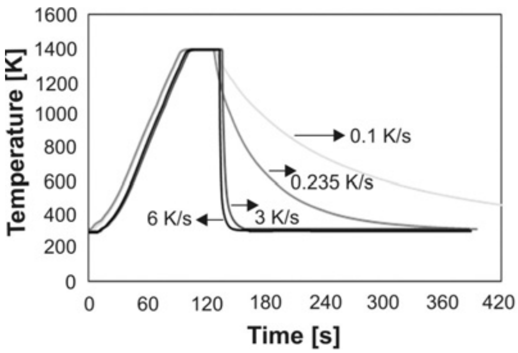
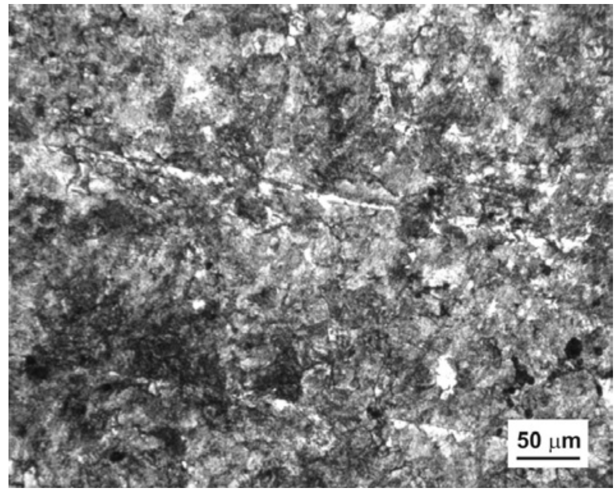


Figure 1. The different vacuum heat treatments with corresponding cooling rates.

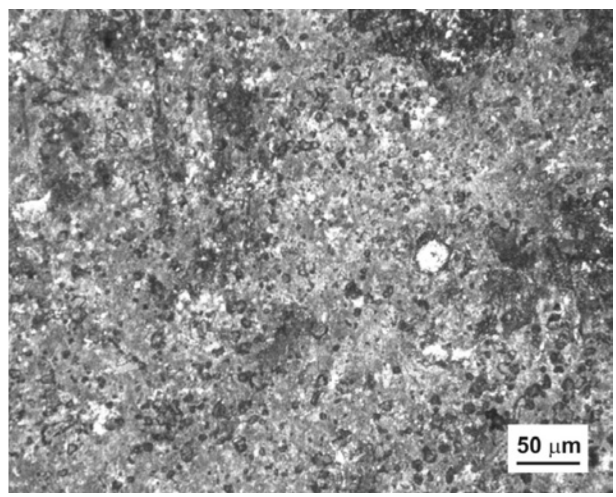
chamber. Different gas pressures were used: 0 Pa, 50 kPa, 200 kPa and 600 kPa. The cooling rate was monitored and recorded by means of thermocouples inserted in the central axis and close to the surface of the specimen. Themocouples placed on the specimen enabled the measurement of the temperature within the specimen core and ensured the exact determination of the cooling conditions. The processes run fully automatically and the documentation of the process results are presented in Figure 1.

The average cooling rates were therefore calculated in the range of 1453 K to 673 K and were 0.1 K/s, 0.235 K/s, 3 K/s and 6 K/s, respectively. The heat treatment conditions consist of the sintering process in vacuum furnace at 1453 K for 1800 s^{-1} with an integrated final tempering at 473 K for 3600 s^{-1}.

Sintered specimens were tested in static tensile tests on a ZWICK Z100 machine, and in an impact testing apparatus ZWICK RKP 450. Microstructures observations were carried out using light microscopy. The apparent hardness HV10 (measured on the tested specimen surfaces) was determined by means of Vickers hardness indenter. Densities were evaluated using the water displacement method.



(a)



(b)

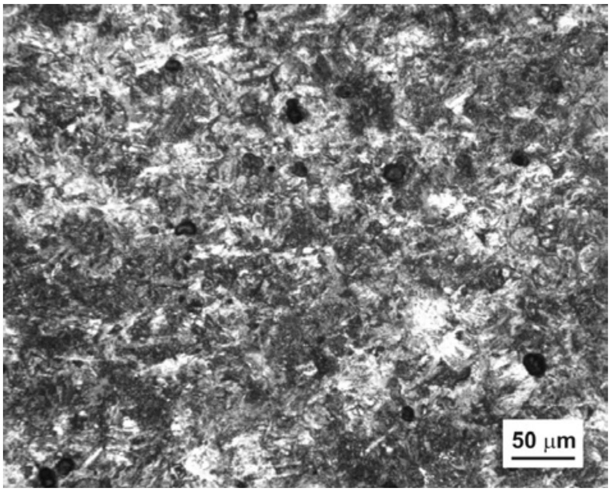
Figure 2. The microstructure cooled at 0.1 K/s (a) and at 0.235 K/s (b).

3 Results and Discussion

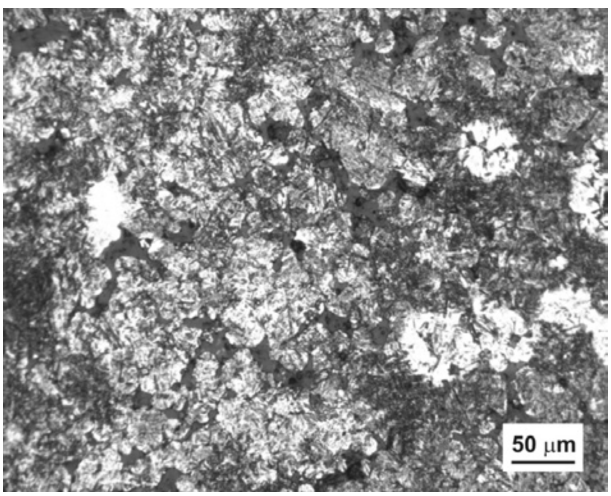
Considering the cooling rates, increasing the nitrogen pressure resulted in an increased amount of bainite/martensite, Figure 2a, b and Figure 3a, b; the microstructure constituents were ranging from 97 % pearlite + 3 % ferite in the system cooled at 0 Pa (0.1 K/s), 77 % bainite + 23 % pearlite in the system cooled at 50 kPa (0.235 K/s), 78 % martensite + 22 % bainite with small amount of tempered martensite in the system cooled at 200 kPa (3 K/s), 82 % martensite + 18 % bainite with small amount of tempered martensite in the system cooled at 600 kPa (6 K/s).

Considering the traditional alloying elements in ferrous PM area, it is clear that chromium and molybdenum represent important alloying elements in term of high strength components included with low cost and environmental benefits. Chromium and molybdenum increase hardenability due to the suppressed effect on the pearlite formation.

Chromium sintered steels offer uniform microstructures ranging between mixture of pearlite and ferrite, full bainite and full martensite [11, 15, 16, 19–22]. Available chemical and microstructural homogeneity of chromium sintered steels can be produced by choosing accurate C-contents and cooling rates. Rapid cooling integrated with sintering may be sufficient to transform a significant portion of the matrix microstructure to martensite, resulting in a significant increase of mechanical properties are called as a sinter hardening [13–16, 23]. At lower cooling rates (in the range of 0.1–0.8 K/s) the microstructure will transform to mainly ferrite, pearlite and/or upper bainite. At higher cooling rates (in the range from 3 K/s) the resulting microstructure will mainly consists of martensite. The fully martensitic mi-



(a)



(b)

Figure 3. The microstructure cooled at 3 K/s (a) and at 6 K/s (b).

crostructures already can be formed using moderate cooling rates (in the range of 1–2.5 K/s) and proper amounts of carbon. To obtain homogenous microstructures after sinter hardening, it is favorable to have a homogenous distribution of the alloying elements already in the compacted part.

The mechanical properties under various processing conditions are summarized in Table 1.

Higher gas pressure increases the martensite content in the microstructure and these results in a further increase in strength with a decrease of ductility and toughness (plasticity properties represent impact energy values).

During the heat treatment, increased alloying elements (chromium, molybdenum and carbon) and/or increased cooling rate cause the austenite to transform to martensite. Martensite is the hardest metallic phase in steel due to carbon remaining in solution and a distortion of the crystal structure to accommodate the carbon.

Conditions	Impact energy	HV10	Transverse Rupture Strength
0 Pa (0.1 K/s)	16.35 J	124.6	763 MPa
50 kPa (0.235 K/s)	11.75 J	161.6	814 MPa
200 kPa (3 K/s)	9.01 J	194.4	940 MPa
600 kPa (6 K/s)	6.97 J	436.8	1000 MPa

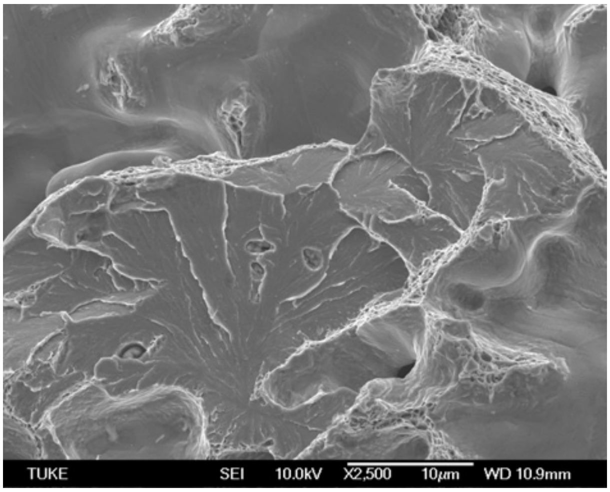
Table 1. Mechanical properties of investigated low alloyed sintered steel.

Hardness has been observed to increase with carbon content only up to 0.5 % C [14]; over this limit, hardness does not increase significantly, but brittleness further rises. Therefore, within certain limits, the cooling rate may help in obtaining a hardened steel with a limited brittleness.

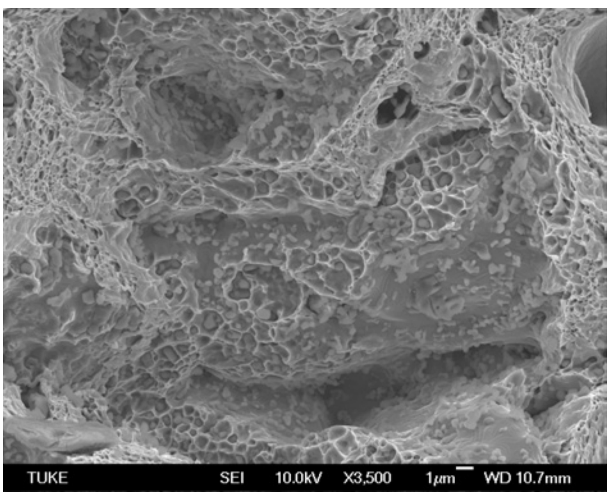
The fracture surfaces of investigated materials are presented in Figure 4a, b. Figure 4a present the cleavage fracture surface with river patterns features. The contents of cleavage rised with increased cooling rates and matches with impact energy data.

The fracture surfaces revealed the huge contaminations (Figure 4b), mainly in places surrounding the original powder particles. The observed contaminations by means of high-resolution SEM coupled with EDX analysis indicate a very high chromium and oxygen content in such contaminations with the presence of some Si and V, indicating that the observed contaminations are complex refractory oxides where chromium oxide is dominant [4, 6, 7]. It is important to note that oxides may form during the atomisation of low alloyed chromium sintered steels. These oxides are much harder to be reduced in subsequent processing. Such a layer of oxides will still cover the surfaces of the annealed powders.

Karlsson et al. [4] revealed that surface oxides on the chromium alloyed sintered steel have a heterogeneous structure consisting of chromium rich particulate compounds (size up to 0.2 μm) surrounded by a thin (6–7 nm) continuous Fe oxide layer. The possibility to reduce these oxides is determined by their thermodynamic stability, which depends on alloy composition of the steel, temperature, and oxygen pressure in the surrounding atmosphere. It is well known that oxides are particularly deleterious to mechanical properties of sintered steels. Regarding heat treatment operations, the main feature of PM steel parts compared to wrought ones is the porosity, and in particular the open one. Danninger and Gierl [24] claimed that the hardenability of sintered steels is lower than that of wrought grades due to the lower thermal conductivity, but this effect should not be overestimated, also with regard to the usually low thickness of PM parts. More problems are connected to the open pores that are filled with media, especially oil during the quenching operation; even by washing opera-



(a)



(b)

Figure 4. The fracture surfaces reveals cleavage features (a) and contamination, mainly oxide, presence (b), cooled at 3 K/s.

tions it is hardly possible to remove this oil from the open pores. Vacuum heat treatment as a one cycle [23], remove the problems related to decarburization and oil (before tempering is necessary to remove it from the open porosity), as well as an inert atmosphere used in sintering process enables to eliminate problem with adequate dew point which it is demand for producing chromium sintered steels and couplet to gas pressure enables in specimens more closed pores; therefore, surface densified layers the removal of surface oxides takes place at the original Fe powder particles.

In any case, vacuum heat treatment can be regarded to offer attractive mechanical properties which can however be accepted for several applications due to improved load-bearing capacity.

4 Conclusions

The obtained results can be summarized as follows:

(i) The microstructures of the investigated sintered specimens were heterogeneous and complex (as function of the gas pressure applied), including different amounts of ferrite, pearlite, bainite and martensite.

(ii) Considering the cooling rates, increasing the nitrogen pressure resulted in an increased amount of bainite/martensite microstructure; the microstructure constituents ranged from 97 % pearlite + 3 % ferite in the system cooled at 0 Pa (0.1 K/s), 77 % bainite + 23 % pearlite in the system cooled at 50 kPa (0.235 K/s), 78 % martensite + 22 % bainite with small amount of tempered martensite in the system cooled at 200 kPa (3 K/s), 82 % martensite + 18 % bainite with small amount of tempered martensite in the system cooled at 600 kPa (6 K/s).

(iii) Vacuum heat treatment supporting bainite-martensitic microstructure with increasing gas pressures, provide a marked increase in strength, nevertheless coupled to a decrease in ductility (mainly represented by values of impact energy) which can however be accepted for several applications.

Acknowledgments

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References

[1] W. B. James: Mater. Des., **8**, No. 4 (1987), 187–197.
[2] J. Bidulská, T. Kvačkaj, R. Bidulský and M. Actis Grande: *High Temp. Mater. Process.*, **27**, No. 3 (2008), 203–207.
[3] P. Ortiz and F. Castro: *Powder Metall.*, **47**, No. 3 (2004), 291–298.
[4] H. Karlsson, L. Nyborg and S. Berg: *Powder Metall.*, **48**, No. 1 (2005), 51–58.
[5] J. Bidulská, R. Kočiško, R. Bidulský, M. Actis Grande, T. Donič, M. Martikán: *Acta Metall. Slovaca*, **16**, No. 1 (2010), 4–11.
[6] E. Hryha, L. Cajkova and E. Dudrova: *Powder Metall. Progr.*, **7** (2007), 181–197.
[7] M. Hrubovčáková, E. Dudrová: *Acta Metall. Slovaca*, **15**, No. 4 (2009), 248–254.
[8] R. Bidulský, M. Actis Grande, M. Kabátová and J. Bidulská: *J. Mater. Sci. Technol.*, **25**, No. 5 (2009), 607–610.
[9] O. Bergman: *Powder Metall.*, **50**, No. 3 (2007), 243–249.

[10] O. Bergman, B. Lindqvist and S. Bengtsson: *Mater. Sci. Forum* (2007), **534–536**, 545–548.

[11] T. Marcu, A. Molinari, G. Straffelini and S. Berg, *Powder Metall.*, **48**, No. 2 (2005), 139–143.

[12] D. Jakubéczyová, M. Hagarová, M. Vojtko: *Acta Metall. Slovaca*, **15**, No. 1 (2009), 15–22.

[13] W. B. James: *Int. J. Powder. Metall.*, **30**, No. 2 (1994), 157–162.

[14] M. Zendron, L. Girardini and A. Molinari: *Powder Metall.*, **51**, No. 3 (2008), 237–244 .

[15] U. Engström, D. Milligan and A. Klekovkin, *Mechanical properties of high performance chromium materials,* Advances in Powder Metallurgy & Particulate Materials, MPIF, San Diego, **vol. 7** (2006), 21–32.

[16] S. Hatami, A. Malakizadi, L. Nyborg and D. Wallin: *J. Mater. Process. Technol.*, **210** (2010), 1180–1189.

[17] E. Dudrová, M. Kabátová, S. C. Mitchell, R. Bidulský, A. S. Wronski: *Powder Metall.*, **53**, No. 3 (2010), 244–250.

[18] E. Dudrová, M. Kabátová, A. S. Wronski, R. Bidulský: *Powder Metall.*, **47**, No. 2 (2004), 181–190.

[19] Anon: Metal Powder Rep., **55**, No. 3 (1999), 22.

[20] J. Bidulská, R. Bidulský, M. Actis Grande: *Acta Metall. Slovaca*, **16**, No. 3 (2010), 146–150.

[21] R. Bidulský, M. Actis Grande J. Bidulská and T. Kvačkaj: *Mater. Tehnol.*, **43**, No. 6 (2009), 303–307.

[22] S. Berg, B. Maroli: *Adv. Powder. Metall. Particul. Mater.*, **8** (2002), 1–14.

[23] R. Bidulský and M. Actis Grande: *High Temp. Mater. Process.*, **27**, No. 4 (2008), 249–256.

[24] H. Danninger and Ch. Gierl: *Int. J. Mater. Prod. Tech.*, **28**, No. 3–4 (2007), 338–360.