Powder Metallurgy Progress, Vol.17 (2017), No.2, p.104-111
http://dx.doi.org/10.1515/pmp-2017-0011

THE EFFECTS OF POST-SINTERING TREATMENTS ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Mn-Mo STEEL

Ch. Fiał

Abstract
The effect of heat treatment on density, hardness, microstructure and tensile properties of Fe-0.85Mo-1.3Mn-0.6C sintered steel were investigated. Pre-alloyed Astaloy 85Mo, ferromanganese and UF4 graphite powders were mixed for 60 minutes in a Turbula mixer and then pressed in single-action die at 660MPa to produce green compacts (according to PN EN ISO 2740).The compacts were sintered in a specially designed semi-closed container at 1120 or 1250°C for 60 minutes in N_2. The chemical composition of the sintering atmosphere was modified by adding getter and/or activator into the container. Two different types of heat treatment in nitrogen were carried out: sinteraustempering at 525°C for 60 minutes; and sinterhardening with additional tempering at 200°C for 60 minutes. The slightly better combination of strength and plasticity of steel for both sintering temperatures were achieved after sinterhardening+tempering variant. Average values of 0.2% offset yield stress, ultimate tensile strength and elongation after sintering in 1250°C, were 415MPa, 700MPa, and 2.0%, respectively.
Kewords: sinteraustempering, semi-closed container, powder metallurgy, mechanical properties, SEM

INTRODUCTION

The production of conventional steel using bainitc quenching with isothermal transformation (austempering) was performed during as early as the 50s of the 20th century [1]. This heat treatment has been and is widely used for combined quenching and tempering. Steels subjected to austempering have higher strength, impact strength and toughness - at a certain hardness - compared to quenched and tempered steels [2, 3]. By combining sintering and austempering in one operation, we can obtain a treatment called sinteraustempering. The steel is cooled rapidly from the sintering temperature to the bainitic region, then it is isothermally annealed to complete the bainitic transformation and subsequently cooled to room temperature – all in one operation. A scheme of sinteraustempering treatment is presented in Fig.1. The purpose of sinteraustempering is also to obtain bainitic steel with a predominance of lower bainite. It is therefore necessary to have knowledge of the temperature at which the bainitic transformation begins. Due to - generally occurring - considerable chemical and structural heterogeneity of sintered steels, it is difficult to speak in general about upper and lower bainite in this case.

Christian Fiał: AGH University of Science and Technology, Cracow, Poland

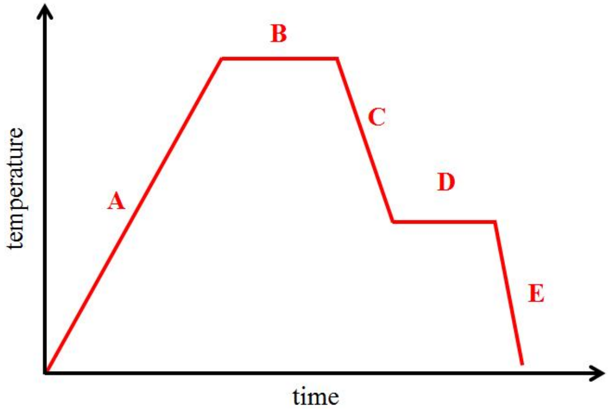

Fig.1. The scheme of sinteraustempering treatment (A – heating, B – sintering, C – rapid cooling, D – isothermal annealing, E – cooling to room temperature)

Despite intensive research and the introduction of austempering into traditional steel manufacturing techniques, its use in powder metallurgy is not widely practiced. There are very few experiments concerning the use of this technique in powder metallurgy. The porosity of sintered steel and its resulting oxidation susceptibility are the main barriers. Moreover, a disadvantage of this process is the relatively high temperature (~500°C) of annealing, encouraging oxidation. Also it is necessary to use a very clean atmosphere with a low content of oxidizing substances like oxygen, water vapour and CO_2 for the correct application of austempering.

Due to the above mentioned reasons, research on sinteraustempering has focused mainly on the use of vacuum furnaces [4] and the use of inert, high pressure blown gas [5, 6]. Unfortunately, the use of vacuum furnaces results in an increase in production costs, which translates into the high price of the produced item. An alternative to sintering steel in vacuum furnaces has been found to be sintering using semi-closed (incompletely sealed) containers, using the effect of the local microatmosphere [7-12]. The effectiveness of this type of solution on a laboratory scale is presented in [13-17].

In this article the results of an exploration of using for sinteraustempering a semi-closed container, with the effect on the local microatmosphere, are presented. For comparison, results of sinterhardening+tempering at200°C are described.

EXPERIMENTAL PROCEDURES

The starting powders were Höganäs Astaloy 85Mo (Fe-0.85%Mo), Elkem II (Fe-77%Mn-1.3%C) and Höganäs C-UF ultra fine pure graphite. Powders were mixed in Turbula mixer for 60 minutes. The nominal compositions are given in Table 1.

Tab.1. Chemical composition of powder mixture (mass %).

Mixture	Fe	Mo	Mn	C
MM	Bal.	0.85	1.3	0.6

The powder mixture were compacted at 660 MPa into standard dog-bone tensile specimens conforming to ISO 2740. Zinc stearate was used for die wall lubrication. Sintering was conducted in a semi-closed stainless steel container, containing a getter and/or activator (to achieve a local microatmosphere), in a laboratory horizontal tube furnace in flowing nitrogen. Two post-sintering variants were then investigated: isothermal annealing at 525° – sinteraustempering (SAT) and rapid cooling from sintering temperature and tempering at 200 °C – sinterhardening (S+H). For each variant 25 specimens were made. The description and designation of those variants are presented in Table 2.

Tab. 2. The description of the post-sintering conditions.

Sintering temperature	Heat treatment type	Time	Designation
1120°C	Isothermal annealing 525°C	60 minutes	SAT_LT
1250°C			SAT_HT
1120°C	Tempering 200°C	60 minutes	SH_LT
1250°C			SH_HT

The sintered samples were tested following standard techniques. Geen and sintered density were determined by water displacement (Archimedes method) after impregnation with paraffin. To examine microstructures, by light microscopy, a Leica DM LM instrument, and by scanning electron microscopy, a FEI Inspect S500 instrument, were employed. Tensile tests were done on a MTS tensile-testing apparatus at a rate of 1mm/min. Furthermore, the three-point bend test on ZD-10 tester using a jig with a span of 28.6 mm, at a crosshead rate of 2 mm/min, was performed. Additionally, for fractography, a Phenom XL instrument was used.

RESULTS

Microstructure

Metallographic investigations were carried out on 3% Nital etched samples. The results of LOM (where **"B"** refers to bainite and **"M"** to martensite) and SEM examinations are presented on Figures 2-9.

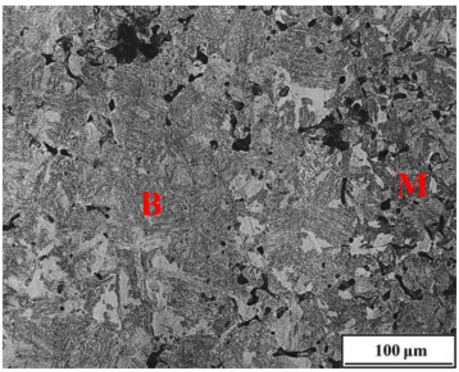

Fig.2. The characteristic microstructure of MM steel after SAT_LT variant (LOM).

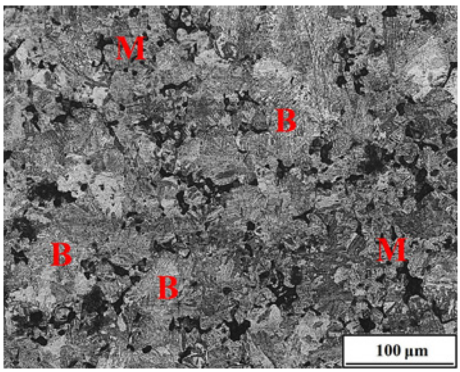

Fig.3. The characteristic microstructure of MM steel after SH_LT variant (LOM).

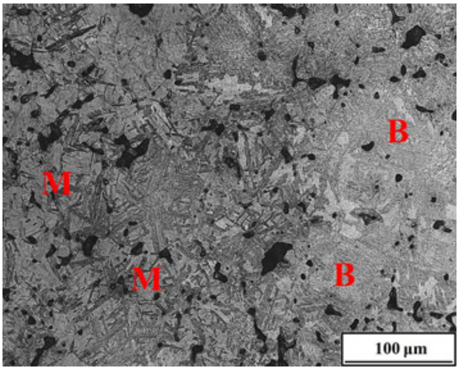

Fig.4. The characteristic microstructure of MM steel after SAT_HT variant (LOM).

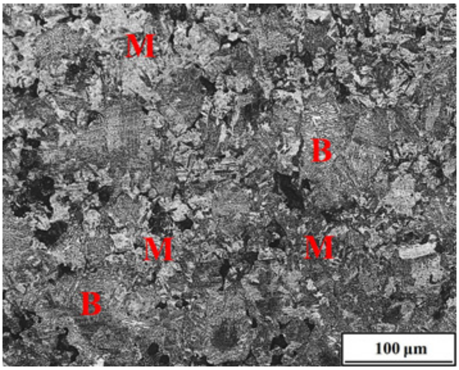

Fig.5. The characteristic microstructure of MM steel after SH_HT variant (LOM).

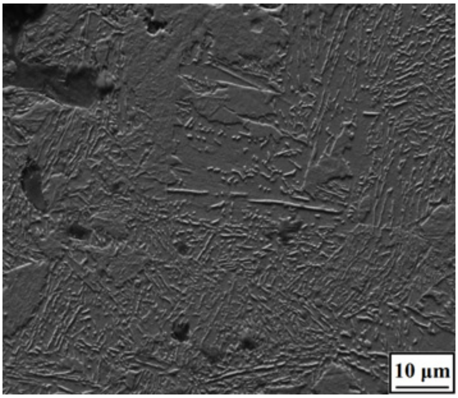

Fig.6. The characteristic microstructure of MM steel after SAT_LT variant (SEM)

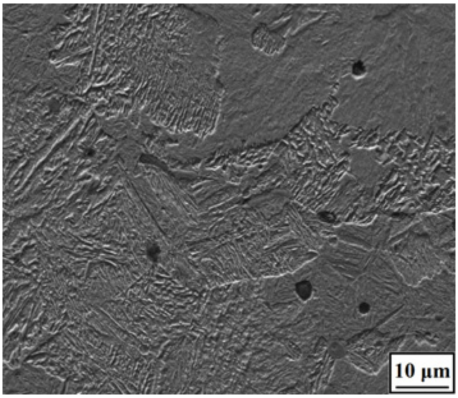

Fig.7. The characteristic microstructure of MM steel after SH_LT variant (SEM)

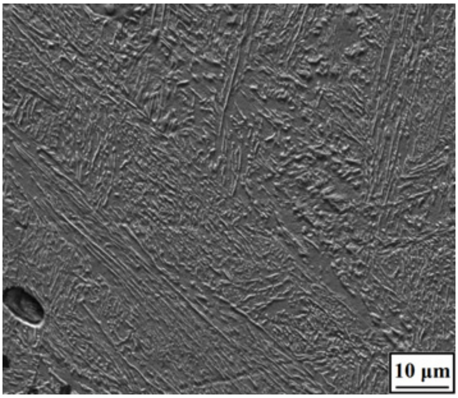

Fig.8. The characteristic microstructure of MM steel after SAT_HT variant (SEM)

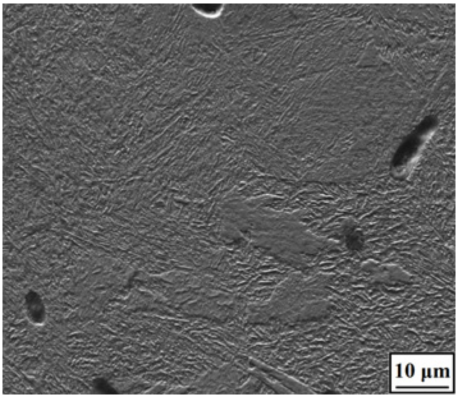

Fig.9. The characteristic microstructure of MM steel after SH_HT variant (SEM)

Regardless of the processing variant and sintering temperature, the microstructures of the tested steels are similar. Both sinteraustempered and sinterhardened steels contain bainite and martensite. However, in the steels obtained by the SH_LT and SH_HT variant, more martensite was observed.

Mechanical properties

The results of density and mechanical testing are listed in Table 3.

Tab.3. Green and as-heat treated densities and mechanical properties of sintered steels – mean values and standard deviations.

Processing Variant	Green density [g/cm^3]	As-heat treated density [g/cm^3]	Yield Strength [MPa]	A [%]	UTS [MPa]	TRS [MPa]
SAT_LT	6.95 ± 0.1	6.97 ± 0.03	353 ± 69	1.4 ± 0.4	577 ± 54	985 ± 92
SH_LT		6.99 ± 0.11	361 ± 55	1.5 ± 0.3	627 ± 59	993 ± 91
SAT_HT		7.06 ± 0.06	400 ± 44	1.9 ± 0.5	647 ± 48	1049 ± 86
SH_HT		7.09 ± 0.05	415 ± 44	2.0 ± 0.3	700 ± 38	1082 ± 132

Density of the steels appears to have increased due sintering at 1250°C, with possibly SH variant being little higher than the sinteraustempering.

Regardless of the processing variant, the differences in properties are not big. Both at 1120°C and 1250°C sintering temperatures, the difference between the UTS of samples obtained by the SH variant and the SAT variant is approximetly 50MPa. The TRS differences between processing variants are even smaller: ~10MPa and ~30MPa for sintering temperatures 1120°C and 1250°C respectively.Both the yield strength and the elongation of the tested compacts, independent of the manufacturing process, are almost at the same level. It is worth to addmit that as the temperature rises from 1120°C to 1250°C, the elongation of the steel test increases by 0.5% and the yield strength by ~ 50MPa. However it should be emphasized, that the values of standard deviations are in some cases greater than the differences obtained between the mean values of steels properties. Therefore, it should be analyzed using statistical techniques (2- and/or 3-parameter Weibull distribution) to be fully sure about any conclusions.

Fractography

The fracture surfaces of the variously processed and tensile tested steels are shown in Figs. 10-13.

The micrographs shown in Fig. 10-11 shows that the fractures of MM steel - sintered at 1120°C and subjected to SAT and SH variants - exhibits ductile character (in an areas of bainite) and small areas with brittle fracture (in an areas of martensite). A similar dependence for the same processing variants is found for compacts sintered at 1250 °C (Figs. 12-13).

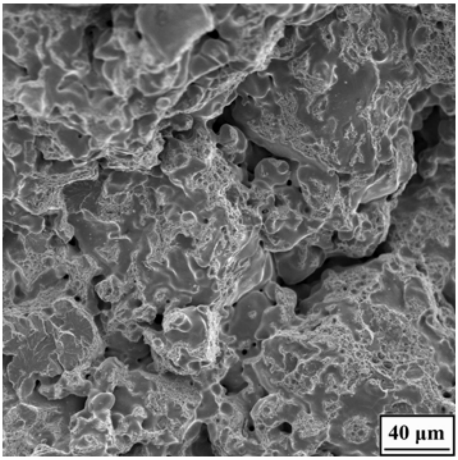

Fig.10. The characteristic fracture of MM steel after SAT_LT variant (SEM).

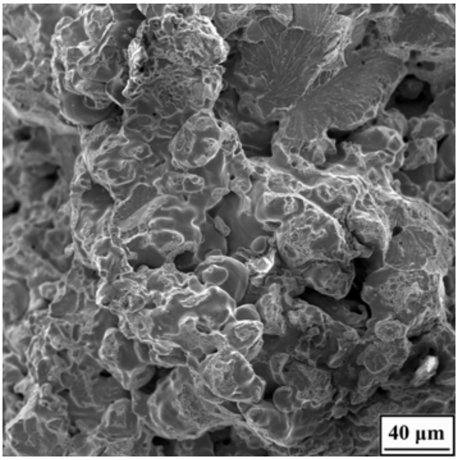

Fig.11. The characteristic fracture of MM steel after SH_LT variant (SEM).

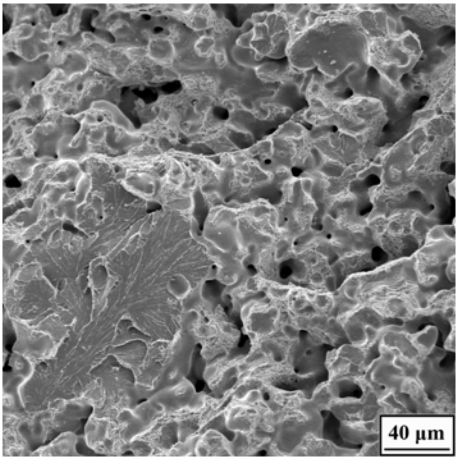

Fig.12. The characteristic fracture of MM steel after SAT_LT variant (SEM).

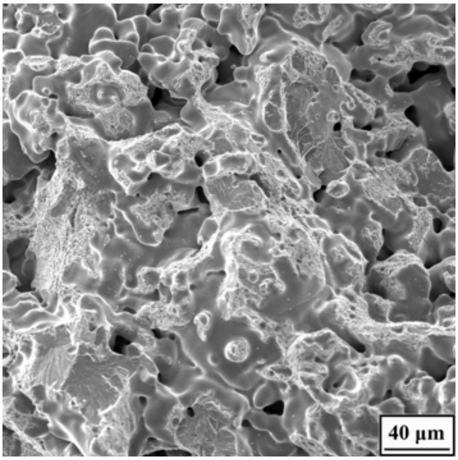

Fig.13. The characteristic fracture of MM steel after SH_LT variant (SEM).

DISCUSSION

The steel examined in this article is a known steel, produced and used in powder metallurgy [18]. Metallographic studies have shown that in compacts subjected to bainitc quenching with isothermal transformation (sinteraustempering), there were small martensite areas along the bainite. The presence of a small amount of martensite in sintered specimens can be associated with too short a time of isothermal annealing (to obtain fully bainitic microstructure) in the range of the bainitic transformation. The same microstructure components were present in the steel samples obtained by the second method (sinterhardening + tempering 200C), but in other proportions - martensite amount increased. Better mechanical properties (perhaps of higher martensite content) and plasticity - regardless of the sintering temperature - were achieved in the case of samples obtained using a large-scale powder metallurgy technique (sinterhardening) and tempered at 200°C. However, the differences between the variants are small (within the standard deviation), so additional calculations using statistical methods (for example, Weibull distribution) should be performed to further characterize the obtained results of steel properties. Strength properties of the obtained steel are slightly better than those of similar chemical compositions mentioned in the literature [19, 20, 21]. The steels presented in this article have much weaker strength than the steels obtained in industry, but they are characterized by higher elongation [18, 22].

CONCLUSION

1. The most important result of these studies is that the semi-closed container processing is a satisfactory technique, on a laboratory scale, for sinteraustempering.
2. MM steel (Fe-1.3Mo-0.85Mo-0.6C) steel was processed successfully, using conventional PM, sinterhardening and/or sinteraustempering experimental procedures. All specimens exhibited ductility,
3. After the sinteraustempering operation at the appropriate temperature, it is possible to obtain a bainitic structure with small areas of martensite in the tested steel.
4. After sinterhardening+tempering at 200°C in the structure of tested steels there was martensite with areas of bainite.
5. The best combination of strength and ductility, regardless the sintering temperature, was recorded for sinterhardening+tempering at 200°C, but the properties of steel obtained by sinteraustempering were only slightly lower.
6. To make better characterization of obtained results of steel properties, the statistical methods should be used.
7. There is the basis for further experiments using semi-closed container and sinteraustempering operation of PM steel.

Acknowledgement

The financial support of the Polish Ministry of Science and Higher Education under the contract No. 11.11.110.299 is gratefully acknowledged.

REFERENCES

[1] Fang, HS., Feng, C., Zheng, YK., Yang, ZG., Bai, BZ.: Journal of Iron and Steel Research, International, vol. 15, 2008, no. 6, p. 1
[2] ASM Handbook. Vol. 4. Metals Park : American Society for Metals, 1991
[3] Metals Handbook. Vol. 2. Metals Park : American Society for Metals, 1964
[4] Girardini, L., Molinari, A., Locatelli, G., Tonini, G.: Metallurgia Italiana, vol. 98, 2006, no. 2, p. 23
[5] Zendron, M., Molinari, A., Girardini, L.: Progress in Powder Metallurgy, vol. 1-2, 2007, no. 534–546, p. 625
[6] Zendron, M., Girardini, L., Molinari, A.: Powder Metallurgy, vol. 51, 2008, no. 3, p. 237
[7] Ciaś, A.: Development and Properties of Fe-Mn-(Mo)-(Cr)-C Sintered Structural Steels. Kraków : AGH - Uczelniane Wydawnictwo Naukowo-Dydaktyczne, 2004
[8] Ciaś, A., Stoytchev, M.: Archives of Metallurgy and Materials, vol. 62, 2017, no. 1, p. 11
[9] Ciaś, A.: International Journal of Materials Research, vol. 106, 2015, no. 5, p. 495
[10] Ciaś, A.: Kovové Materiály, vol. 54, 2016, no. 4, p. 269
[11] Ciaś, A.: Powder Metallurgy, vol. 56, 2014, no. 3, p. 231
[12] Ciaś, A., Mitchell, SC., Pilch, K., Ciaś, H., Sułowski, M., Wroński, AS.: Powder

Metallurgy, vol. 46, 2003, no. 2, p. 165
[13] Fiał, C., Dudrová, E., Kabátová, M., Kupková, M., Selecká, M., Sułowski, M., Ciaś, A.: Powder Metallurgy Progress, vol. 14, 2014, no. 3, p. 137
[14] Fiał, C., Dudrová, E., Kabátová, M., Kupková, M., Selecká, M., Sułowski, M., Ciaś, A.: Powder Metallurgy Progress, vol. 15, 2015, p. 124
[15] Fiał, C., Dudrová, E., Kabátová, M., Kupková, M., Selecká, M., Sułowski, M., Ciaś, A.: Archives of Metallurgy and Materials, vol. 60, 2015, no. 2, p. 783
[16] Fiał, C., Ciaś, A., Czarski, A., Sułowski, M.: Archives of Metallurgy and Materials, vol. 61, 2016, no. 3, p. 1201
[17] Fiał, C., Ciaś, A., Sułowski, M.: Naučni Izvestiâ na Naučno-tehničeskiâ S'ûz po Mašinostroene, vol. 24, 2016, no. 1, p. 256
[18] Hoeganes Corporation Data Sheet: ANCORBOND FLM Alloys
[19] Selecká, M., Simkulet, V. In: PM2004 Powder Metallurgy World Congress & Exhibition, Vienna. Shrewsbury : EPMA, 2004, p. 237
[20] Selecká, M., Simkulet, V., Keresti, R., Parilák, L. In:EURO PM2005 Congress & Exhibition, Prague. Vol. 1. Shrewsbury : EPMA, 2005, p. 41
[21] Dudrová, E., Kabátová, M., Bidulský, R., Wroński, AS.: Powder Metallurgy, vol. 47, 2004, no. 2, p. 181
[22] James, WB., Lindsley, B., Narasimhan, KS.: Powder Metallurgy Progress, vol. 12, 2012, no. 1, p. 3