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The Microstructure and Properties Evolution in Fe–(1.5-2.5)%Mn-0.8%C Systems during Step Sintering

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Abstract
The aim of the study was to examine how the microstructure changes during heating of Fe-Mn-C system (step-sintering). Mixtures of powders containing Fe (NC 100.24 iron powder), 1.5 or 2.5%Mn (added in the form of ferromanganese – 77%Mn, 1.3%C) and 0.8%C (added in the form of ultrafine graphite) were prepared in Turbula T2F mixer for 30 minutes.
For the investigation of microstructural evolution of Fe-Mn-C system, from each chemical composition, batches of 5 or 10 samples were prepared in accordance to PN EN ISO 2740 standard. Single-action pressing at 660 MPa resulted in green densities in the range of 6.53-6.60 g/cm^3.
The procedure of investigations was as follows: samples were heated to the different step temperature (varied from 750°C to 1250°C) with heating rate 60 °C/min., then isothermally sintered at step temperature for 5 min., and finally cooled to the room temperature with cooling rate ~66 °C/min.
After step-sintering, Fe-Mn-C samples were mechanically (tensile) tested. Following tensile tests, metallographic observations of the samples were performed. Based on the results obtained, the tensile strength increasing with the increasing the step temperature. The metallographic observation showed the microstructure evolution – with increasing the step temperature, decreasing of porosity was observed.

Keywords: powder metallurgy, step sintering, manganese, microstructure

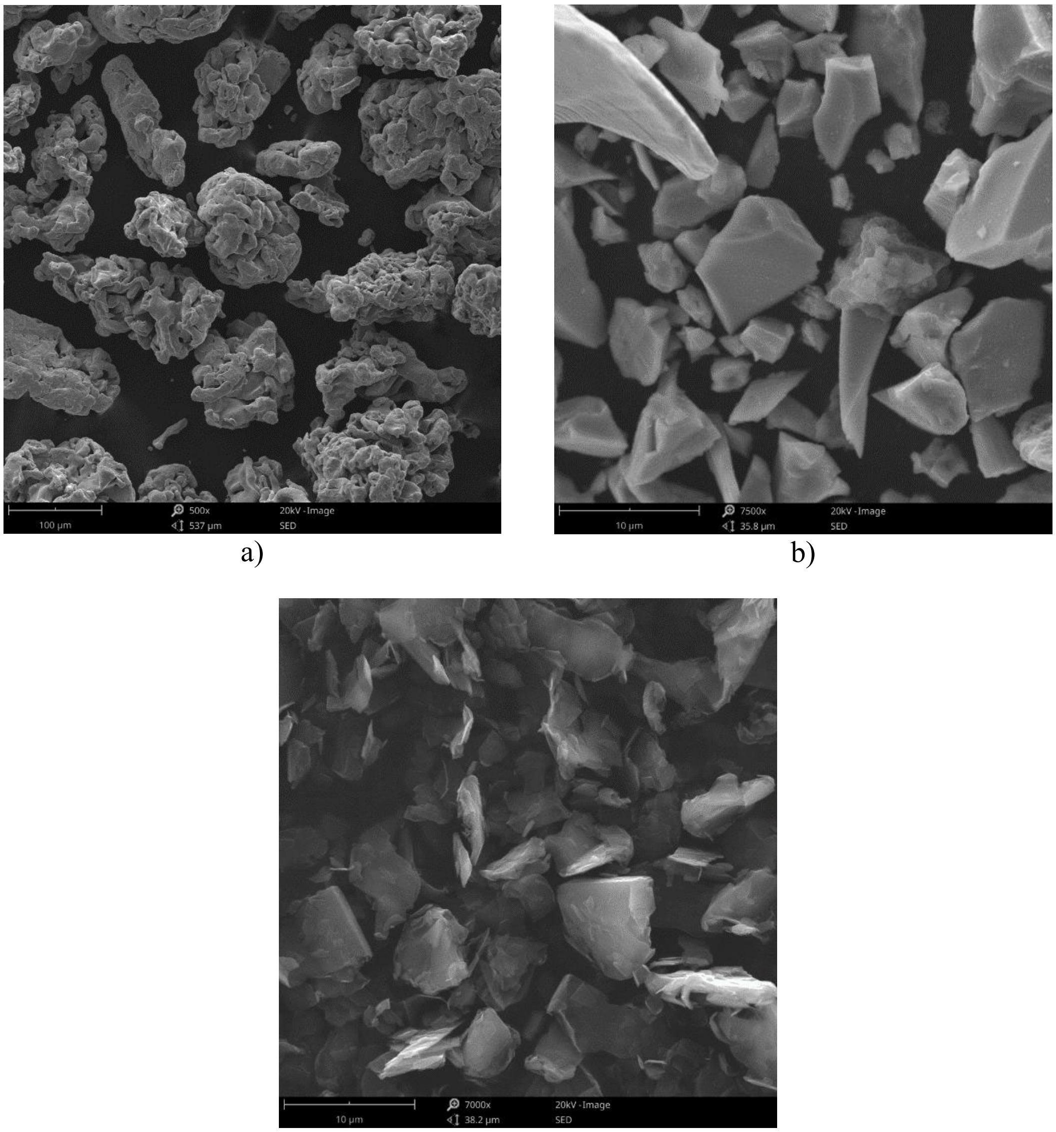
1. Introduction

Steels produced by powder metallurgy (PM) route are now increasingly used in the industry due to the arbitrary selection of chemical compositions, often impossible to produce by other methods. [1-6]. This is related to the use of a sintering temperature lower than the melting point of the main component. Additionally, during PM processing, there is almost no material lost and the temperatures used for steel processing are lower than during casting, which positively affects the economic aspect of the PM process. The evolution of microstructure influences the material properties. This is why the proper selection of chemical composition and production conditions is so important. In the case of sintered the Fe-Mn-C steels, manganese, being an austenite stabilizing element, shifts the ES (A_{cm}) line to the left to the lower carbon content, and the GS line to lower temperatures [7-11]. The increase of manganese content causes the formation of a wider three-phase area: ferrite, austenite and manganese cementite [7,8].
The occurrence of austenite in the structure causes the presence of different microstructural constituents – pearlite, bainite and/or martensite – depending on the local manganese content in the sintered steel and cooling rate. Previous studies of sintered steels containing 1 to 3% Mn [11] showed that with increasing Mn content in steel up to 2.5%, the strength properties increased (UTS up to 754 MPa and TRS up to 1187 MPa) together with persistent high plastic properties (A = 3.66% and YS = 623 MPa). The microstructure of 2.5%Mn PM steels consisted mainly of pearlite and bainite. After exceeding the Mn content up to 2.5%, the microstructure of steel was martensitic which strongly caused reducing the plasticity of the material. Manganese is an alloying element with high affinity to oxygen which resulted in the occurrence

of manganese oxides at the sintered steel. This is the main reason in decreasing its strength properties with increasing Mn content.

2. Experiments

The following starting powders were used: Höganäs iron powder grade NC 100.24, Elkem ferromanganese powder (77%Mn, 1.3%C) and ultra-fine (C-UF) graphite powder (Fig. 1).



a) b)

c)

Fig. 1. The starting powders: a) Höganäs iron powder grade NC 100.24, b) Elkem ferromanganese powder and c) ultra-fine (C-UF) graphite powder

The average particle size was 106 µm, below 30 µm and 9 µm for NC 100.24, Elkem and graphite powders, respectively. Oxygen content for NC100.24 was 0,21 mass% and for Elkem 0,91 mass%. From starting powders, the mixtures with the composition Fe-X% Mn-0.8% C

(X = 1.5 and 2.5) were prepared in Turbula T2F mixer for 30 minutes. To determine the step temperatures, dilatometric investigations were earlier carried out [12].
For the investigation of microstructural evolution of Fe-Mn-C system, from each chemical composition, batches of 5 or 10 samples were prepared in accordance to PN EN ISO 2740 standard (Fig. 2).

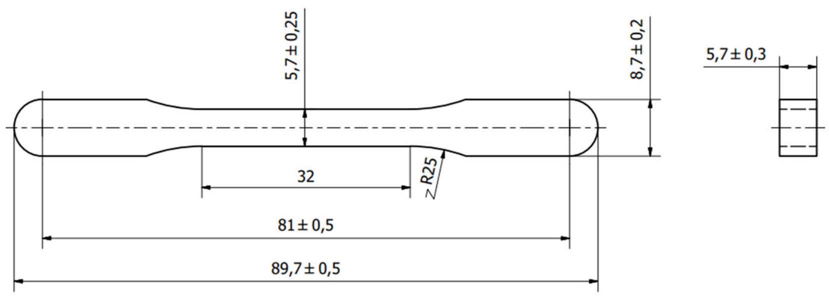


Fig. 2. Test piece for tensile testing

Single-action pressing at 660 MPa resulted in green densities at the level of approx. 6.68 g/cm^3. After pressing, green compacts were heated (heating rate 60 K/min.) to step temperature (Table 1), sintered in the atmosphere consisted of 95%N_2-5%H_2 for 5 minutes and cooled with cooling rate ~66K/min. Finally, samples were mechanically (tensile strength and Vickers hardness) tested and metallographic observations were performed.

3. Results

The results of tensile strength tests and hardness of investigated materials are summarized in Tables 1 and 2 and Figs. 3 and 4, respectively.

Table 1. Ultimate tensile strength, UTS, elongation after tensile test and $R_{0,2}$ yield offset of investigated Fe-Mn-C materials

Chemical composition	Sintering temperature [°C]*	UTS [MPa]	A [%]	$R_{0.2}$ yield offset [MPa]
Fe-1.5%Mn-0.8%C	750	86±20	0.4±0.2	84±19
	760	109±10	0.9±0.2	90±8
	770	112±11	0.4±0.1	111±11
	800	114±5	0.8±0.2	97±3
	820	131±5	0.4±0	131±5
	850	145± 4	0.5±0.1	143±2
	860	140±13	1.5±0.2	110±10
	870	159±22	0.6±0.2	149±16
	900	146±2	1.1±0.3	122±15
	920	200±12	0.8±0.2	184±4
	970	244±15	2.8±0.4	160±9
	1030	315±9	4.7±0.6	171±7
	1120	482±10	4.3±0.5	264±20
	1250	596± 30	5.8±1.2	283±7
Fe-2.5%Mn-0.8%C	750	77±10	0.3±0.1	N/D
	760	66±9	0.22±0	N/D
	770	94±8	0.4±0.0	N/D
	800	81±11	0.24±0.1	N/D

	820	141± 10	0.5±0.1	138±8
	850	139±26	0.5±0.1	135±23
	860	129±13	0.46±0.1	N/D
	870	173±14	0.8±0.2	152±7
	900	153±27	0.6±0.2	139±9
	920	229±9	1.1±0.2	183±7
	970	292±19	1.9±0.2	173±11
	1030	385±20	3.8±0.4	164±6
	1120	497 ±24	3.6±0.3	276±14
	1250	638±9	5.0±0.4	272±10

*cooling rate- ~1K/s

Table 2. Vickers hardness of investigated Fe-Mn-C materials – mean of 5 measurements

Chemical composition	Sintering temperature [°C]	HV 0.025	Chemical composition	Sintering temperature [°C]	HV 0.025
Fe-1.5%Mn-0.8%C	750	139±10	Fe-2.5%Mn-0.8%C	750	145±22
	770	140±20		770	143±13
	820	142±17		820	162±12
	850	147±16		850	164±29
	870	149±20		870	165±32
	920	171±12		920	197±20
	1120	243±30		1120	260±16
	1250	315±51		1250	324±60

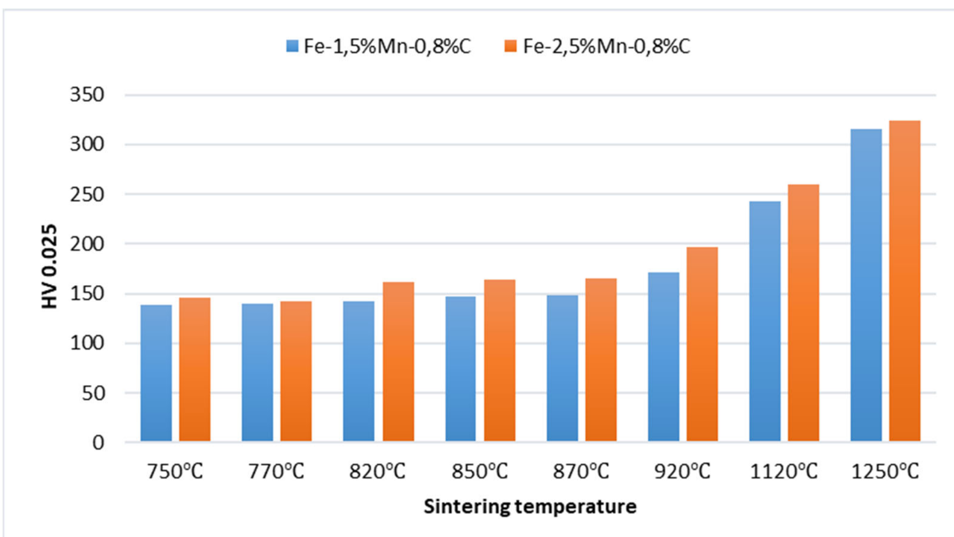
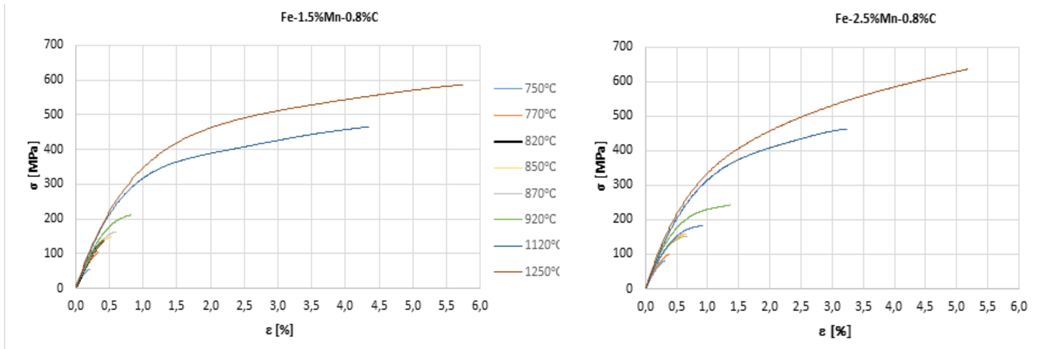


Fig. 3. Vickers hardness of investigated materials vs. sintering temperature

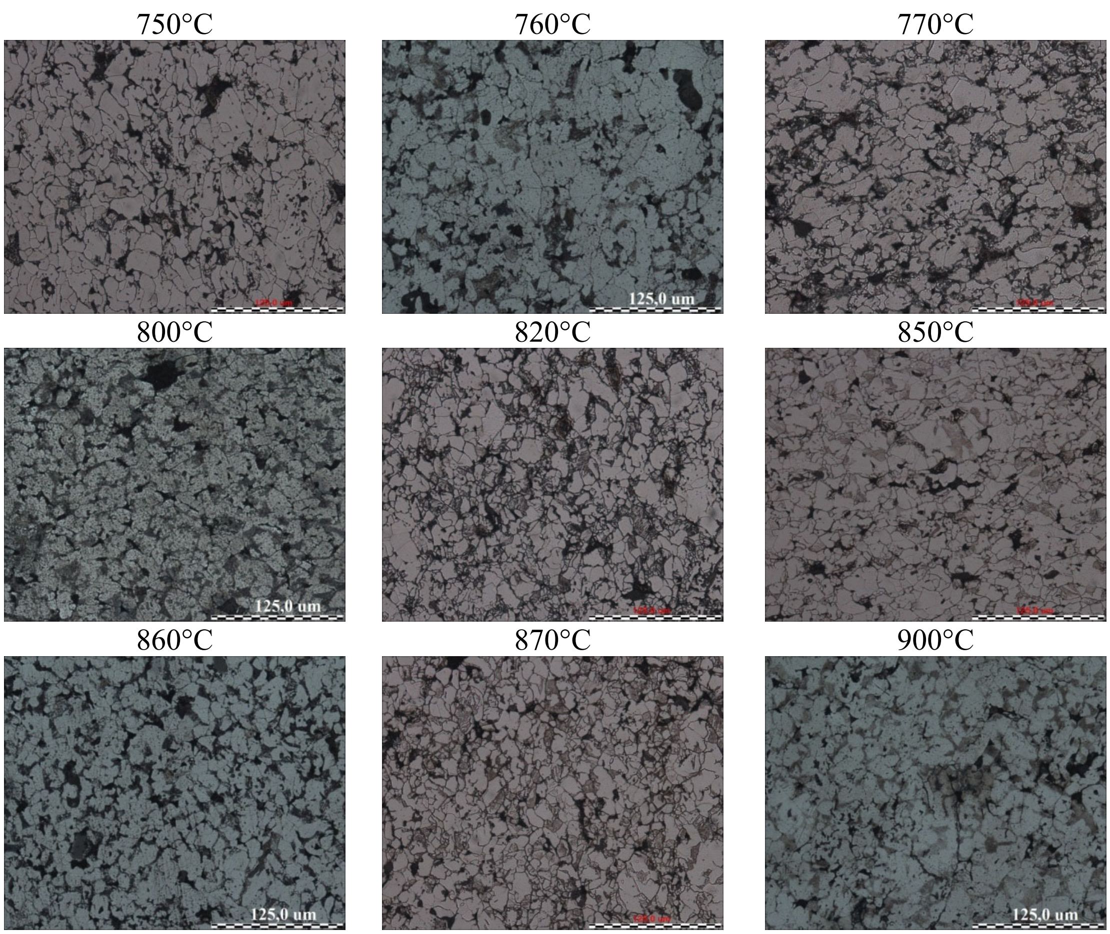
When analyzing the tensile curves of the tested materials, presented in Fig. 4, it can be seen that their nature changed with increasing sintering temperature. Moreover, these curves can be divided into two groups. The first one consists of materials sintered at lower temperatures, from 750 ° C to 920°C, for which the tensile strength was in the range of 86 MPa – 200 MPa and 77 MPa – 229 MPa, respectively for the manganese content of 1.5% and 2.5%. The elongation after the tensile test of these materials did not exceed 1.5% (Figs. 4a, 4b). The second group of tensile curves are those recorded for steel sintered at 1120°C and 1250°C. These materials reached the tensile strength of 482 MPa – 596 MPa (Fe-1.5% Mn-0.8% C steel) and 497 MPa – 638 MPa (Fe-2.5% Mn-0.8% C steel). The steels sintered at the temperature of 1250°C, regardless of the manganese content, were characterized by elongation exceeding 5%.



a) **b)**

Fig 4. The effect of sintering temperature on the tensile strength of a) Fe-1.5%Mn-0.8%C and b) Fe-2.5%Mn-0.8%C

The effect of step temperature on the microstructural changes of Fe-Mn-C materials was investigated on samples etched by 3% Nital (Figs. 5 and 6) using Leica DM4000M microscope.



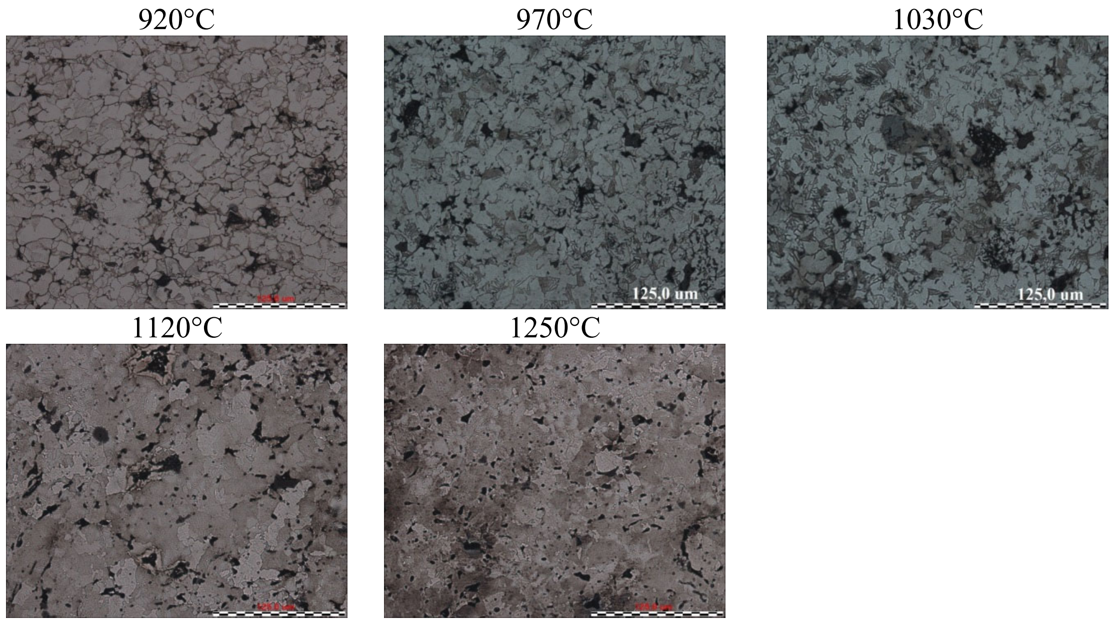
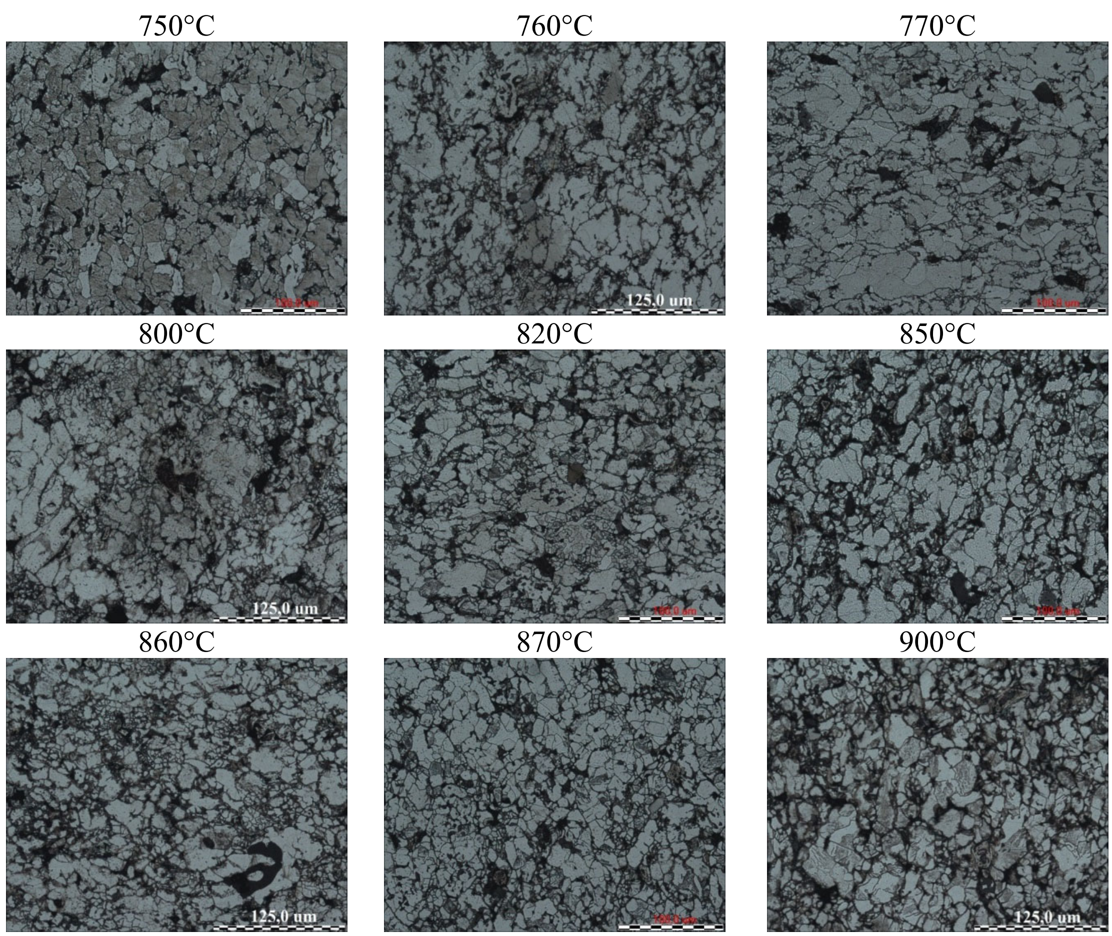


Fig. 5. The microstructure of Fe-1.5%Mn-0.8%C vs. annealing temperature; mag. 200x



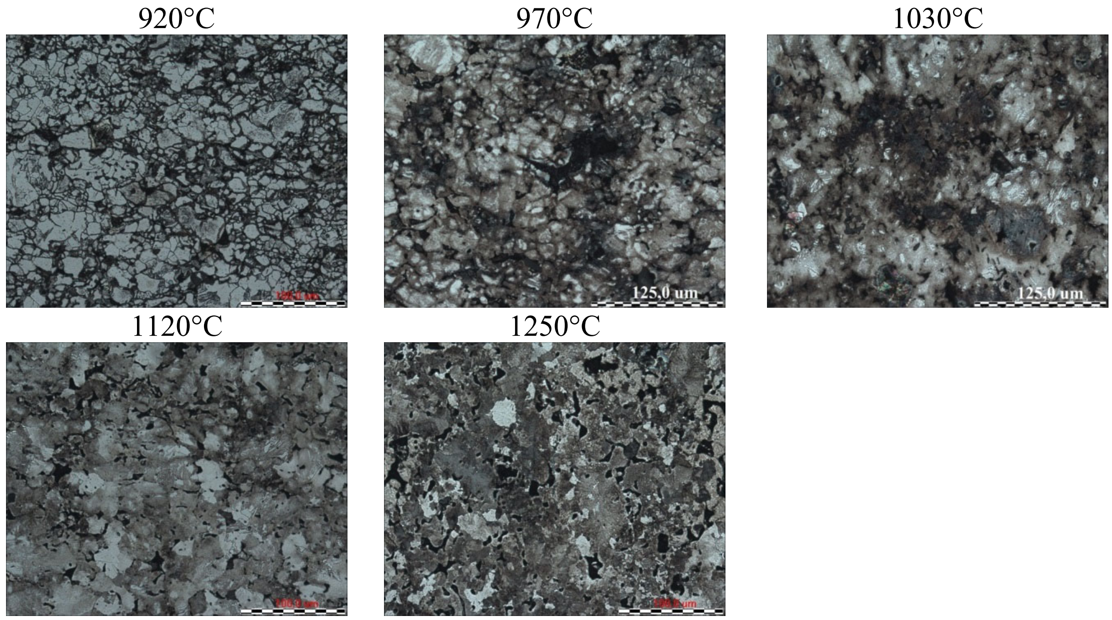


Fig. 6. The microstructure of Fe-2.5%Mn-0.8%C vs. annealing temperature; mag. 200x

Investigations of the microstructure of sintered Fe-Mn-C steels showed their heterogeneity. There were mainly two structural components in the tested steels: ferrite and perlite. The steel having a chemical composition of 2.5% Mn, after sintering at 1030°C, was characterized by a pearlitic microstructure. This was due to the fact that manganese lowers the temperature of the eutectoid transformation. As a result, the pearlitic transformation could fully take place at a lower temperature. Moreover, when analyzing the phase equilibrium diagram of Fe-Mn, it can be noticed that manganese, dissolving in iron, lowers the transformation temperature of Fe-α to Fe-γ (ferrite to austenite). The carbon present in the steel dissolves in austenite, which in turn turns into pearlite when the steel is cooled. The share of individual microstructural components depended on the manganese content and the sintering temperature.

Analyzing the results of metallographic tests, it can be concluded that the share of individual structural components of the tested materials changes with increasing sintering temperature. Steels with the composition Fe-1.5%Mn-0.8%C, sintered at the temperature of 750°C, were characterized by a ferritic microstructure. Undissolved ferromanganese particles are also visible in the microstructure of these materials. Perlite grains appear after sintering at 770°C. As the sintering temperature increases, the volume fraction of perlite increases. The steel sintered at the temperature of 1120°C was characterized by a complex, ferritic-pearlitic structure dominated by fine perlite. Its further heating to the temperature of 1250°C contributed to obtaining a pearlitic microstructure with a small proportion of ferrite. The development of the microstructure of the Fe-2.5%Mn-0.8%C steel was similar. In the microstructure of steel containing 1.5% manganese, sintered at 1120°C and 1250°C, martensite could be observed, which may indicate that the steel is hardening.

Because manganese was added to the powder mixture in the form of ferroalloy, the deteriorating compressibility of the samples was observed and, as a consequence, after annealing at the step temperatures, higher porosity. The occurrence of larger pores contributed to the decreasing in strength properties after sintering at temperatures close to the α-γ transformation, e.g. at temperature of 750°C UTS decreasing from 107 MPa to 71 MPa and 30 MPa, for samples containing 1.5%, and 2.5% Mn, respectively.

The results obtained after the step-sintering test confirmed the research [5] where it was determined, that for sintered steel containing 0.8% C, the most favorable strength properties were obtained for 2-2.5% Mn. Further increase in Mn content up to 3% caused decreasing of the strength properties. Metallographic examinations confirmed the α-γ transformation temperatures, determined during dilatometric investigations. For Fe-1%Mn-0.8%C steel, sintered in the range from 750°C to 850°C, after cooling, mainly homogeneous, ferritic structure was observed. The exceptions were the places of insoluble ferromanganese particles, significantly lowering the austenitizing temperature and, as a consequence, contributed to the formation of perlite in the structure. For higher manganese contents, pearlitic regions were observed after sintering at 820°C (for 2% Mn) and 750°C for 3% Mn. After sintering at 1120°C and 1250°C, the microstructure of steels was mainly pearlitic-ferritic with the bainite areas for 3% Mn. Too short sintering time was not enough to fully austenitize sintered steel containing 3% Mn and thus to form martensitic structure, what was observed in such steels after sintering for 60 minutes [11].

4. Summary

During step sintering tests it was possible to observe the evolution of the microstructure of Fe-Mn-C system. Additionally, for Fe-Mn-C materials, the effect of Mn addition, added in the form of ferroalloy, on formability, and in the consequence, of the sinterability of investigated steels was observed. The study also showed that after sintering of Fe-Mn-C steel for only 5 minutes, slightly lower properties were obtained than after sintering during 60 minutes. Following the results obtained, following conclusions can be drawn:

1. The sintering temperature did not affect the as-sintered density.
2. Steels containing 2.5% of manganese were characterized by lower density.
3. The mechanical properties of sintered Mn steels increase with increasing sintering temperature.
4. The Fe-1.5%Mn-0.8%C and Fe-2.5%Mn-0.8%C steels, sintered at 1250°C, were characterized by high tensile strength, which allows them to be classified into the group high-strength steel (600 – 1000 MPa).
5. The highest elongation, at the level of 5.8%, was recorded for the Fe-1.5% Mn-0.8% C steel sintered at 1250°C.
6. The sintering time has no major influence on the mechanical properties of sintered manganese steels. Sintering for 5 minutes. at a temperature of 1120°C or 1250°C, it allows to obtain steel with satisfactory properties.
7. The sintering temperature in the range of 770°C – 920°C did not have a significant influence on the microhardness of sintered manganese steels. Significant differences in the results of microhardness were observed for the temperatures of 1120°C and 1250°C, which is related to an increase in the microstructure of the tested materials of pearlite.
8. The steels Fe-1.5% Mn-0.8% C and Fe-2.5% Mn-0.8% C had a heterogeneous microstructure.
9. With the increase of the sintering temperature, the amount of perlite in the microstructure of sintered manganese steels increased.

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References

1. Danninger H. et al.: Powder Metallurgy, vol. 48, (2005), 23-32.
2. Hryha E. and E., Dudrova, In: Application of Thermodynamics to Biological and Materials Science, vol. 22. InTech, Rijeka, Croatia, 573–602.
3. Hryha E., E., Dudrova, Mater. Sci. Forum, vol. 534-536, (2007), 761-764.
4. Dudrova E. et al., Powder Metallurgy, vol. 53, (2010), 244-250.
5. Gomez-Acebo M. et al, Computer Coupling of Phase Diagrams and Thermochemistry, vol. 27, 2003, 325-334.
6. Hryha E. et al, Journal of Materials Processing Technology, vol. 212 (2012), 977– 987.
7. Staub F., Metaloznawstwo, Śląskie Wydawnictwa Techniczne. Katowice. 1994.
8. Desforges C. D., W. E. Duckworth, T. F. J. N. Ryan, Manganese in ferrous metallurgy, Edited by The Manganese Centre, ISBN 2 901 109 004.
9. Massalski T. B., Binary alloy phase diagrams, American Society for Metals. Ohio. 1986.
10. Malkiewicz T., Metaloznawstwo stopów, Państwowe Wydawnictwa Naukowe. Łódź. 1978.
11. Tenerowicz-Żaba M., M. Sułowski, A comparison of mechanical properties and microstructures of PM steels with chemical compositions Fe-(1-3)%Mn-0.8%C, Science of Sintering, vol. 50, No 4, (2018), 457-466.
12. Tenerowicz-Żaba M., M. Sułowski, Step-sintering of Fe-(1-3)%Mn-0.8%C steels, Materials Science Forum, to be published