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THE EFFECTS OF PROCESSING PARAMETERS ON THE STRUCTURE AND MECHANICAL PROPERTIES OF STRUCTURAL PM STEELS CONTAINING Mn, Cr AND Mo

WPŁYW PARAMETRÓW WYTWARZANIA NA STRUKTURĘ I WŁASNOŚCI MECHANICZNE SPIEKANYCH STALI KONSTRUKCYJNYCH ZAWIERAJĄCYCH Mn, Cr I Mo

The effects of processing parameters on the microstructure and mechanical properties of Fe-Mn-Cr- Mo-C PM steels are described. Pre-alloyed Astaloy CrM and Astaloy CrL, low-carbon ferromanganese and graphite powders were used as the starting materials. After pressing in rigid die, the compacts were conventionally and high temperature sintered at 1120 and 1250°C, respectively. Sintering was carried out for 60 minutes in atmospheres with different H_2/N_2 ratios. Cooling rate from sintering temperature was 65°C min^{-1} (convective cooling). The specimens were subsequently tempered at 200°C for 60 minutes in air. All specimens were tested for tensile strength (UTS), elongation (A), offset yield strength ($R_{0.2}$), transverse rupture strength (TRS), impact toughness and apparent surface hardness (HV 30). After mechanical tests the microstructure of Fe-Mn-Cr-Mo-C PM steels was studied by optical microscopy. These investigations have shown that, by sintering in inexpensive and safe nitrogen-rich atmospheres, it is possible to achieve mechanical properties similar to those of specimens sintered in pure hydrogen and hydrogen-rich atmospheres.

Keywords: powder metallurgy, processing parameters, mechanical properties, sinterhardening

W pracy przedstawiony został wpływ parametrów wytwarzania na mikrostrukturę i własności spiekanych stali Fe-Mn-Cr-Mo. Do badań użyto wstępnie stopowanych, komercyjnych proszków żelaza Astaloy CrM i Astaloy CrL, proszek żelazomanganu oraz proszek grafitu. Po sporządzeniu mieszanek proszków, wypraski były prasowane jednostronnie w sztywnej matrycy, a następnie spiekane w temperaturze 1120°C i 1250°C, w atmosferze o różnym składzie chemicznym (różny stosunek H_2/N_2 w atmosferze spiekania) w czasie 60 minut. Po spiekaniu próbki poddane zostały zabiegowi odpuszczania w powietrzu, w temperaturze 200°C w czacie 60 minut.

Spieczone i odpuszczone próbki poddane zostały badaniom mechanicznym (R_m, R_g, HV 30) oraz badaniom metalograficznym z wykorzystaniem mikroskopii optycznej.

Z przeprowadzonych badań i uzyskanych rezultatów wynika, że własności spiekanych stali manganowo-chromowo-molibdenowej, wytwarzanych przy wykorzystaniu taniej i bezpiecznej atmosferze bogatej w azot są zbliżone lub niekiedy nawet wyższe od własności stali spiekanych w atmosferach bogatych w wodór.

1. Introduction

Powder metallurgy (PM) steels are differ from their wrought counterparts. This technique is very often used for the production of sintered iron-base parts, mainly alloyed with Ni and Cu. Because of high price of Ni and its cancerogenic effect [1, 2], as well as problems with recycling copper-containing scrap-metal, these alloying elements can be substituted by other safe and cheaper elements, for example Mn, Cr and Mo. These promote the formation of hard phases such as bainite and martensite and they are very commonly used in high-strength sintered structural steels. Chromium, manganese (both having high affinity for oxygen) and molybdenum all enhance hardenability. Consideration of Ellingham-Richardson diagrams [3] indicates that sintering of manganese steels is not practicable in endogas and, even in pure hydrogen, the dew point requirements for sintering temperatures of 1120 and 1250°C are -60°C and -50°C, respectively.

Manganese enhances hardenability, but its concentration must be kept to a minimum to avoid blocky regions of retained austenite. The influence of manganese and nitrogen sintering atmosphere on the mechanical properties of PM steels is reported in numerous papers [4], while the results reporting the simultaneous influence of Cr, Mo and Mn are limited [5-10]. Molybdenum also enhances resistance to temper embrittlement. Mo additions are more effective than additions of Mn, their oxides are reducible during sintering in standard industrial conditions (1120°C, dissociated ammonia atmosphere, -30°C dew point).

Sintered steels are very often heat treated, when quenching and tempering is the standard route in the production of tough PM steels. One interesting alternative, avoiding heat

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TABLE 1

Green and as-sintered densities of Fe-Mn-Cr-Mo-C PM steels

Chemical composition	Green density	As-sintered density		Green density	As-sintered density	
	ISO 2740 bars			rectangular 5×10×55 mm bars		
		1120°C	1250°C		1120°C	1250°C
	d_1, g/cm^3	d_2, g/cm^3		d_1, g/cm^3	d_2, g/cm^3	
CrL+3Mn+0.3C	6.92	6.91	6.91	7.14	7.08	7.18
CrL+3Mn+0.8C	6.81	6.81	6.94	6.95	6.95	7.14
CrM+3Mn+0.3C	6.86	6.84	6.88	7.06	7.00	7.09
CrM+3Mn+0.8C	6.87	6.88	6.99	7.08	7.01	7.32

treatments, to get the same or improved mechanical properties is to generate bainitic microstructures by continuous cooling transformation (sinterhardening). Therefore bainitic Mn-Cr-Mo sinterhardened steels are being considered as possible candidates to replace the traditional PM steels [11]. Steels like these are found to transform into virtually fully bainitic microstructures with very little martensite by cooling from standard sintering temperature with typical cooling rates [4]. It becomes difficult to adopt this route when the strength required is in excess of 500 MPa whilst maintaining toughness, but there has been progress in the context of continuously cooled low-alloy structural PM steels.

The second alternative heat treatment, which is currently underdeveloped, is sinteraustempering. The idea of this treatment is to cool steels from the sintering temperature down to a temperature in which bainitic transformation take place, isothermally annealing in this temperature, and then cooling to room temperature. The first attempt of using sinteraustempering was made by Molinari [12].

The aim of this work was to study the effect of processing parameters on the structure and mechanical properties of PM steels containing Mn, Cr and Mo.

2. Experimental methods, materials and procedures

Two pre-alloyed Höganäs Astaloy CrL and Astaloy CrM powders were used as the basis materials. 3% of manganese, in the form of low-carbon (1.3% C) ferromanganese (77% Mn) powder, 0.3% and 0.8% carbon, in the form of ultra fine graphite powder, were added to the base powders in order to prepare Fe-3Mn-1.5Cr-0.2Mo-(0.3/0.8)C or Fe-3Mn-3Cr-0.5Mo-(0.3/0.8)C (based on pre-alloyed Astaloy CrL and Astaloy CrM, respectively) mixtures by blending the components in a Turbula mixer for 30 minutes. The mixed powders were then compacted in steel dies with zinc stearate lubricated walls. Three types of compacts were prepared: 5×10×55 mm TRS specimens, ISO 2740 dog-bone tensile test bars and 4×4×15 mm dilatometric bars (green densities of about 6.4-6.5 g/cm^3). The green densities (d_1) of the compacts are summarised in Table 1.

Isothermal sintering was carried out in dry (10 ppm moisture) atmospheres with different H_2 to N_2 ratios (Table 2), in a laboratory horizontal tube furnace at 1120 and 1250°C for 60 minutes, employing convective (65°C/min) cooling, as was suggested in Refs. [13, 14]. In the sinterhardening process, cooling control technique is important for achieving target strength and toughness. To improve the local dew point of the microatmosphere and to minimise the loss of manganese due to volatilisation, sintering was carried out in a semi-closed stainless steel container. The as-sintered densities (d_2) of the sintered compacts are summarised in Table 1. Sintering of dilatometric bars was carried out in a horizontal push rod dilatometer NETZSCH 402E. The measuring direction (length of the specimens) was chosen perpendicular to the pressing direction of the compacts. Pure, dry hydrogen, nitrogen and mixture of 5%H_2-95%N_2 with a dew point below -60°C and with a flow rate of about 10 ml/min were used as the sintering atmospheres. Heating and cooling rates were 10°C/min and 20°C/min, respectively. Isothermal sintering was carried out for 60 min. at two temperatures: 1120°C and 1250°C. As-sintered densities of samples were in the range of about 6.5-6.6 /cm^3.

TABLE 2

Metallic radii of rare earth metals and magnesium [12]

Atmosphere code	Chemical composition of sintering atmosphere
A0	H_2
A1	75H_2 / 25 N_2
A2	25 H_2 / 75 N_2
A3	5H_2 / 95 N_2
A4	N_2

After sintering, the set of 5 pairs of samples was tempered at 200°C for 60 minutes in air. The mechanical properties of the steels are given in Tables 3 and 4. After mechanical testing, the structure of the steels was examined by optical microscopy and SEM techniques. LECO instruments were employed to measure the chemical compositions of the PM steels.

3. Results

3.1. Mechanical properties and microstructure

The physical and mechanical properties of Fe-Mn-Mo-Cr-C PM steels are summarised in Tables 1, 3 and 4. An increase in the Cr content from 1.5 to 3 wt.-% was observed to result in clearly higher strength levels. The apparent hardness and strength increase with Cr content for all sintering atmospheres. The TRS and UTS of the 3%Cr alloy are significantly higher than for the 1.5% Cr-containing alloys. Both alloys showed a strong effect of the sintering temperature on impact toughness.

During the investigations, a decarburisation effect was observed. The higher carbon loss, of 0.11%, was recorded for CrM samples sintered at 1250°C in A3 atmosphere with H_2/N_2 ratio 3:1. The measured carbon content for the samples was found to be 0.190-0.304 wt.-%C (Leco CS 125). There was a little effect of Cr content on oxygen carbon. The hardness of the alloys sintered at 1120 and 1250°C is shown basically to be a function of carbon content. Oxygen content (Leco TC 336),which can often be a problem in Cr-Mn containing PM steels, could be reduced below 0.2 wt.-% with high temperature sintering. Higher oxygen contents (0.310-0.548 wt.-%) were measured at conventional sintering temperatures (Table 5). There was visible effect of Cr content on oxygen content. The data presented in Table 5 indicate that increasing the chromium content increases the carbon concentration in the sintered steel. Because the A2 atmosphere (25%H_2-75%N_2) is not very often used during processing PM steels, the chemical composition of steels was investigated only for A0, A1, A3 and A4 atmospheres.

The data presented in Table 6 indicate that the loss of carbon is observed in steels sintered in A0 (pure hydrogen) atmosphere, and this effect is decreasing with increase of the nitrogen content in the sintering atmosphere. The nitriding effect is observed in specimens sintered in nitrogen-rich atmospheres – the nitrogen level increase up to 10 times.

TABLE 3

Mechanical properties of Fe-3Mn-(Cr)-(Mo)-0.3C PM steels

Chemical composition / sintering atmosphere / sintering temperature			UTS, MPa	$R_{0.2}$ yield offset, MPa	A, %	TRS, MPa	Impact toughness, J/cm^2	HV30
CrL+3Mn+0.3C	A0	1120°C	554	481	0.8	1216	3.37	245
	A1		439	–	0.6	1017	3.04	262
	A2		662	608	1.0	1049	3.55	318
	A3		542	481	0.8	1020	3.04	284
	A4		541	481	0.8	1191	3.40	274
CrM+3Mn+0.3C	A0	1120°C	439	357	0.6	1195	5.82	305
	A1		563	–	0.8	1030	3.33	268
	A2		645	560	1.1	1105	4.95	317
	A3		648	537	1.7	982	3.34	323
	A4		706	535	2.5	855	4.05	320
CrL+3Mn+0.3C	A0	1250°C	604	474	1.2	1613	15.18	222
	A1		682	475	1.5	1549	10.93	244
	A2		753	537	1.6	1348	6.47	278
	A3		785	535	1.7	1407	5.96	273
	A4		829	554	2.0	1469	5.78	272
CrM+3Mn+0.3C	A0	1250°C	811	594	1.8	1543	6.55	302
	A1		752	632	1.3	1665	8.12	313
	A2		905	613	2.3	1695	6.88	304
	A3		872	615	2.2	1610	8.92	319
	A4		954	562	2.8	1658	10.71	315

TABLE 4

Mechanical properties of Fe-3Mn-(Cr)-(Mo)-0.8C PM steels

Chemical composition / sintering atmosphere / sintering temperature			UTS, MPa	$R_{0.2}$ yield offset, MPa	A, %	TRS, MPa	Impact toughness, J/cm^2	HV30
CrL+3Mn+0.8C	A0	1120°C	474	440	1.53	1100	3.79	302
	A1		495	–	0.98	1020	4.65	246
	A2		468	–	1.18	959	3.60	326
	A3		429	417	1.22	955	3.50	314
	A4		431	–	1.18	924	3.34	343
CrM+3Mn+0.8C	A0	1120°C	431	405	1.36	791	3.50	374
	A1		466	–	1.56	911	5.47	426
	A2		461	426	1.51	836	5.38	489
	A3		413	–	1.27	877	4.33	459
	A4		389	360	1.31	910	4.51	413
CrL+3Mn+0.8C	A0	1250°C	704	583	3.29	1316	8.25	109
	A1		593	–	2.09	1336	6.63	261
	A2		667	459	2.44	1299	5.73	382
	A3		680	487	2.59	967	5.14	367
	A4		684	432	2.58	1178	5.24	403
CrM+3Mn+0.8C	A0	1250°C	887	536	4.56	1305	7.71	439
	A1		669	517	2.39	1174	6.28	301
	A2		791	586	3.46	1169	8.70	456
	A3		654	560	2.31	1352	8.61	469
	A4		671	517	2.58	1274	8.63	473

TABLE 5

Oxygen and carbon contents of PM Fe-3Mn-(Cr)-(Mo)-0.3C steels

Sintering temperature, °C and atmosphere		CrL+3Mn+0.3C		CrM+3Mn+0.3C	
		C, wt.-%	O_2, wt.-%	C, wt.-%.	O_2, wt.-%
1120	A0	0.287	0.587	0.303	0.507
	A1	0.266	0.331	0.277	0.310
	A3	0.289	0.460	0.304	0.390
	A4	0.274	0.338	0.276	0.333
1250	A0	0.221	0.252	0.218	0.343
	A1	0.208	0.140	0.221	0.161
	A3	0.190	0.157	0.228	0.290
	A4	0.191	0.165	0.214	0.280

The microstructure of convective (65°C/min) cooled and tempered PM steels are shown in Figs. 1-2. The primary microstructural constituent of the two alloys is bainite. Figure 1 shows the microstructures of the alloys sintered at 1120°C. Both of the Cr-Mn containing alloys have similar structures, which are predominantly bainitic with a small percentage of martensite and austenite. The microstructure of bainitic steel is more complex than that of pearlitic steel and is largely dependent on the composition and processing conditions. The bainitic microstructures reveal a mixture of tempered martensite and bainite associated with intralath carbides. The tempered martensite is distinguished from the bainite by the precipitation of multiple, rather than single, cementite structures within each lath.

TABLE 6

Oxygen and carbon contents of PM Fe-3Mn-(Cr)-(Mo)-0.8C steels

Sintering temperature, °C and atmosphere		CrL+3Mn+0.8C		CrM+3Mn+0.8C	
		C, wt.-%	O_2, wt.-%	C, wt.-%.	O_2, wt.-%
1120	A0	0.645	0.313	0.707	0.594
	A4	0.724	0.569	0.770	0.587
1250	A0	0.525	0.497	0.615	0412
	A4	0.599	0.398	0.618	0.379

In sinterhardened PM steels containing sufficient quantities of Cr and Mn, it is possible to obtain a microstructure consisting of a mixture of bainite, carbon enriched retained austenite, and only some martensite. The mechanism by which bainite grows, places strict limits on the temperature range for transformation and on the maximum amount of transformation that can be achieved. The blocks of austenite (Figs. 1-2) tend to transform to high carbon, untempered martensite under the influence of small stresses and consequently have an embrittling effect. The essential principles governing the optimisation of such microstructures are well established, particularly that large regions of unstable high-carbon retained austenite, known to be detrimental for toughness, must be avoided.

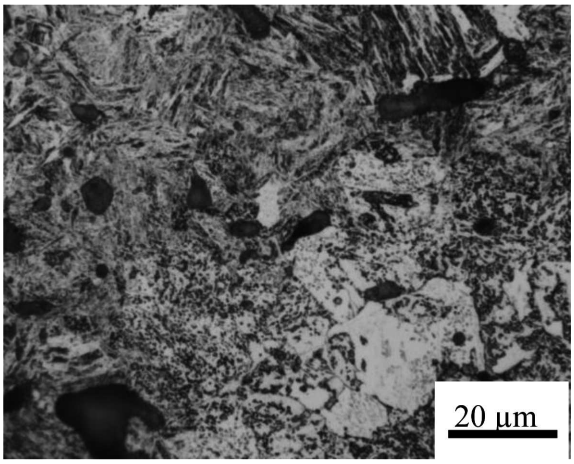

Fig. 1. Micrograph of Astaloy CrL-based PM steel sintered at 1120°C in H_2, tempered at 200°C for 60 min; bainite, martensite and of blocky austenite islands

The developed steels had a microstructure which is classified as a medium-carbon bainitic/martensitic. In these steels bainite consists of an aggregate of acicular ferrite and carbides. Its morphology changes progressively with the transformation temperature, i.e. grain size and acicularity of the structure increase as the temperature decreases. Upper bainite comprises larger ferrite plates, bounded by Fe_3C precipitates that form directly from the austenite. As shown in Figs 1-2, specimens with lower Cr and Mo contents, sintered at 1120°C, show a bainitic/martensitic structure, whereas in those containing more Cr and Mo, martensitic/bainitic/austenitic structures are mainly observed. After sintering at 1250°C, the structure consists mainly of martensite and bainite regions with small amount of austenite.

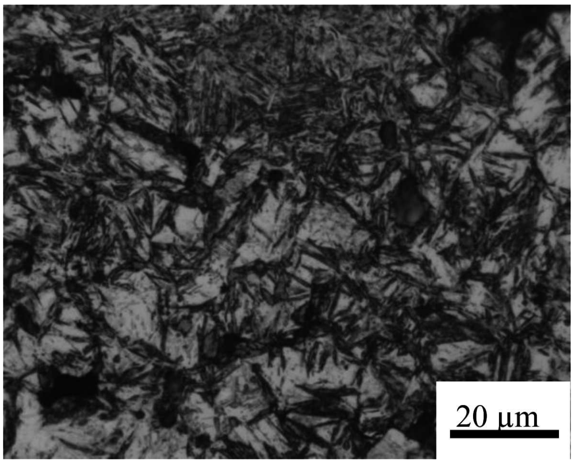

Fig. 2. Micrograph of Astaloy CrM-based PM steel sintered at 1120°C in N_2, tempered at 200°C for 60 min; bainitic microstructure embedded in a carbon-enriched matrix of austenite

3.2. Dilatometric investigations

The results of dilatometric investigation of Fe-3%Mn-(Cr)-(Mo)-(0.3-0.8)%C PM steels are presented in Tables 7 and 8 and Figure 3. During isothermal sintering, shrinkage was recorded in the range of -0.30% to -1.70% and -0.32% to -1.11% for CrM- and CrL-based materials, respectively. Total dimensional changes are higher for specimens with addition of 3 mass % Mo and 1.5 mass % Cr. Taking into account the effect of sintering atmosphere on dimensional stability, there is no clear relation between shrinkage/swelling

TABLE 7

The temperatures of M_s and B_s for Fe-3%Mn-(Cr)-(Mo)-(0.3-0.8)%C PM steels sintered at 1120°C and 1250°C

Chemical composition	Atmosphere	Sintering temperature 1120°C			Sintering temperature 1250°C		
		C, mass %	B_s, °C	M_s, °C	C, mass %	B_s, °C	M_s, °C
CrL+3Mn+0.3C	A0	0.3	475	300	0.3	300	250
	A3		475	300		300	250
	A4		400	275		300	250
CrM+3Mn+0.3C	A0	0.3	500	300	0.3	400	200
	A3		400	ND		300	ND
	A4		300	ND		275	ND
CrL+3Mn+0.3C	A0	0.8	300	200	0.8	200	100
	A3		200	150		200	100
	A4		200	125		175	75
CrM+3Mn+0.3C	A0	0.8	400	ND	0.8	375	ND
	A3		300	ND		275	ND
	A4		175	ND		200	ND

and chemical composition of the sintering atmosphere. The $\alpha \rightarrow \gamma$ transformation in these PM steels is in the range of 790°C – 850°C and no effect of chemical composition of sintering atmosphere on this transformation was recorded. A small effect of Cr and Mo content on the $\alpha \rightarrow \gamma$ transformation was recorded.

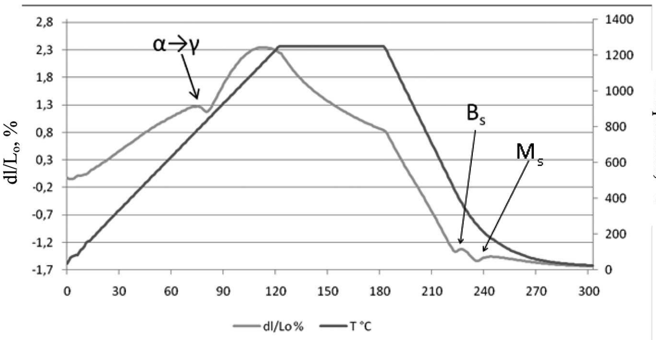

Fig. 3. Dilatometric curve of sintered Fe-3%Mn-1.5%Cr-0.2% Mo-0.8%C PM steel. This curve schematically presents the way of calculating the M_s and B_s temperature

As is presented in Table 7, an effect of carbon concentration on M_s and B_s was observed. It could be explained by a favourable effect of carbon on the hardenability of steels. Also the M_s and B_s temperatures depend on sintering temperature; increasing the sintering temperature, generally decreases the temperature of bainite and martensite transformations.

4. Discussion

An investigation has been made of the microstructures and mechanical properties of 20 series of sintered Fe-Mn-Cr-Mo-C steel specimens. These alloys can be sintered over the entire range of temperatures typically used for iron-based PM alloys. By optimizing alloying elements, it is possible to obtain, after continuous cooling transformations, a bainitic microstructure that has a small cooling rate dependence. The high bainite content results in the alloys having high hardness and yield strength as compared to other high performance PM ferrous alloys.

The investigated alloys belong to the group of low alloyed sinterhardened medium-to-high strength steels, which are used for structural parts in ferrous powder metallurgy and with success can substitute traditional, expensive PM steels. The recorded tensile and yield strengths of investigated steels were up to 887 and 586 MPa, respectively.

The mechanical properties of investigated PM steels appear comparable to other studies [4, 14-16]. A comparison of the tensile strength, elongation and impact values is found to be in quite good agreement. The effect of using a higher sintering temperature than 1120°C on mechanical properties is evident from the presented results. The tensile and transverse rupture strengths data show a measurable effect of the sintering temperature. The application of semi-closed container offers a means to increase the mechanical properties of single compacted steels to those typical of double pressed and sintered steels.

The work was carried out using N_2/H_2 atmospheres with a dew point - 60°C (~20 ppm moisture), so it is rather difficult to make a direct comparison with results obtained in industry, and with other published work, where different N_2/H_2 atmospheres had been used. The dew point refers to the dryness of a H_2 only atmosphere; if diluted (given water vapour content), the reducing potential is decreased, because amount of H_2 is reduced. In flowing N_2 atmosphere reduction of MnO oxides by solid carbon below 1425°C is impossible. Only control of the local microclimate in semi-closed container, i.e. CO/CO_2 ratio, ensures optimum conditions for carbothermic oxide reduction and efficient sintering [4]. Specimens sintered at 1250°C possessed higher mechanical properties, irrespective of the H_2/N_2 ratio in the furnace atmosphere. Chromium enhances, however, as compared with previous results [15], the detrimental effects of N_2 on the strength of the manganese steels. This effect can be confirmed by chemical analysis in the investigated steels.

The addition of chromium and manganese improves the alloy hardenability, producing a virtually fully bainitic/martensitic microstructure in the sinterhardened condition. However, sinterhardening of Fe-Mn-Cr-Mo-C steels requires careful process control over the entire manufacturing process. It is clear that there is no one single key important part of the processing route. By understanding the intertwined relationship between the processing route and the physical metallurgy, it is possible to develop a range of low alloy Mn-Cr steel grades.

Regarding microstructures, N_2 with a dew point of -60°C did not prove as successful a furnace atmosphere as equally dry H_2; a furnace atmosphere of dry N_2 is not as effective in preventing formation of deleterious oxide networks as of dry H_2, or of H_2-N_2 mixtures.

The developed steels can replace quenched and tempered PM steels and make it possible to reduce energy consumption and costs in the manufacturing process of structural components. According to recent results, low alloy Fe-Mn-Cr-Mo-C bainitic steels show good strength-toughness balance, and, unlike conventional non sinter-hardened steels, they can achieve strengths that is equal to those of quench-tempered low alloy steels.

5. Conclusions

This study has contributed to showing the possibility of sintering Fe-Mn-Cr-Mo-C steels in chemically different atmospheres. On the basis of present work, the following conclusions can be drawn:

1. The significance of the development of the sinterhardened PM Mn-Cr bainitic steel can be summarized as follows: (a) good combination of strength and toughness; (b) self-hardening with high bainitic hardenability by convective cooling (65°C/min) from the sintering temperature without additional quenching/tempering treatment; (c) reducing costs of both raw materials and production; and (d) savings in energy resources.
2. It is possible to sinter Mn and Mn-Cr-Mo steels in other than a hydrogen-rich atmosphere without decreasing their mechanical properties. The mechanical properties are satisfactory, in contrast to those achieved after sintering in hydrogen-rich atmospheres.

3. Investigated steels belong to the medium-to-high strength sintered steels.
4. The optimized chemical composition, alloying technique and processing of investigated PM steels result in high mechanical properties of the material, which can be improved by increasing sintering temperature and/or applying sinterhardening. The need for a secondary quench-hardening treatment is eliminated.
5. Increasing the nitrogen content in the sintering atmosphere decreases the B_s and M_s temperatures.
6. Using nitrogen as a sintering atmosphere increases surface hardness of Fe-Mn-Cr-Mo-C steels.
7. Alloying elements improve the hardness of steel and decrease the temperature of bainite and martensite transformations.
8. Sintered steels based on Astaloy CrM pre-alloyed powder can be called self-hardened steels because of the low cooling rate necessary to obtain bainitic/martensitic or martensitic structure.

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