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SINTERED STRUCTURAL PM Cr AND Cr-Mo STEELS

SPIEKANE STALE KONSTRUKCYJNE CHROMOWE I CHROMOWO-MOLIBDENOWE

The object of the study was the evaluation of the effect of production parameters on the microstructure and mechanical properties of Cr and Cr-Mo PM steels. The steels were processed from commercial Höganäs pre-alloyed powders: Astaloy CrA, Astaloy CrL and Astaloy CrM with carbon, added in the form of grade C-UF graphite powder in amounts of 0.4 and 0.8 wt. %. Following Turbula mixing for 30 minutes, green compacts were single pressed at 660 MPa according to PN-EN ISO 2740 standard. Sintering was carried out in a laboratory horizontal furnace at 1120°C and 1250°C for 60 minutes in a 5%-95% hydrogen-nitrogen atmosphere. After sintering, the samples were tempered at 200°C for 60 minutes in air. Mechanical tests indicate that the steel based on Astaloy CrA pre-alloyed powder could be an alternative material to steels based on Astaloy CrM. Steels sintered at the higher temperature revealed better mechanical properties.

Keywords: sintered steels, alloying elements, mechanical properties, microstructure

Celem pracy była ocena wpływu parametrów wytwarzania na strukturę oraz własności mechaniczne spiekanych stali konstrukcyjnych. Badania przeprowadzono na spiekanych stalach, wykonanych na bazie komercyjnych, stopowych proszków żelaza Höganäs: Astaloy CrA (1,8% Cr, 98,2% Fe), Astaloy CrL (1,5% Cr, 0,2% Mo, 98,25% Fe), Astaloy CrM (3% Cr, 0,5% Mo, 96,5% Fe) z dodatkiem proszku grafitu C-UF. Zawartość węgla w mieszance proszków wynosiła 0,4% i 0,8%. Mieszanki proszków prasowano jednostronnie w stalowej matrycy pod ciśnieniem 660 MPa, otrzymując kształtki zgodne z PN-EN ISO 2740. Sprasowane kształtki poddano spiekaniu w temperaturze 1120°C i 1250°C w czasie 60 minut. Atmosferę spiekania stanowiła mieszanina wodoru i azotu w ilości 5%H_2-95%N_2. Po spiekaniu kształtki były odpuszczane w temperaturze 200°C przez okres 60 minut.

Przeprowadzone badania własności mechanicznych wykazały, że stale wykonane na bazie proszku stopowego Astaloy CrA, mogą stanowić alternatywę dla spieków wykonanych na bazie proszku stopowego Astaloy CrM, a ponadto spieki wytworzone w wyższej temperaturze wykazywały lepsze własności mechaniczne.

1. Introduction

Structural material parts in construction of machines can be made by PM technology. These include cogwheels, piston rings, compressor wings, even space shuttle skin plating, and household objects. Over 90% of world sintered material produced is based on iron [1-3]. The manufacturing process of PM technology consists usually of four stages: production of powders, their mixing, forming and sintering. Very often after sintering, a final treatment is used to achieve better properties of the sintered materials [3]. Sintered materials based on pure iron have low mechanical properties, so there is a need to use some additives for improving their properties. The most popular additive is carbon, added in the form of graphite in the maximum amount of 0.3% [4]. According to the carbon content, several microstructures can result: ferrite-pearlite, pearlite or pearlite with pro-eutectoid cementite [2, 5]. Tensile strength of carbon steels does not exceed 520 MPa, and to obtain higher properties, it is necessary to introduce alloying elements such as Cu and Ni [6-11]. However, problems with recycling scrap metal containing copper and the high price of nickel result in the substitution of these elements in sintered steels by Mn and Cr [4, 9, 12-14]. Such Mn or Mn+Cr sintered steels attain higher mechanical properties in comparison with Cu and Ni steels. What is more, they can be produced with lower costs [10, 11, 14-28].

2. Experimental methods, materials and procedures

Pre-alloyed Höganäs iron powder: Astaloy CrL (1.5% Cr, 0.2% Mo, 98.25% Fe), Astaloy CrM (3% Cr, 0.5% Mo, 96.5% Fe) and Astaloy CrA (1.8% Cr, 98.2% Fe) were used as the base powders. Carbon, in form of graphite C-UF (ultra fine), in amounts of 0.4% and 0.8% was introduced into the powder mixes (Table 1), which were Turbula mixed for 30 minutes. Physical and technological properties of the powders were reported in Ref. 29.

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TABLE 1

Chemical composition and description of powder mixtures

Chemical composition	Designation	Alloyed iron powder	Cr, wt. %	Mo, wt. %	C, wt. %	Fe
Fe+1.8%Cr+0.4%C	CrA+0.4%C	Astaloy CrA	1.8	–	0.4	Bal.
Fe+1.5%Cr+0.2%Mo+0.4%C	CrL+0.4%C	Astaloy CrL	1.5	0.2	0.4	Bal.
Fe+ 3%Cr+0.5%Mo+0.4%C	CrM+0.4%C	Astaloy CrM	3	0.5	0.4	Bal.
Fe+1.8%Cr+0.8%C	CrA+0.8%C	Astaloy CrA	1.8	–	0.8	Bal.
Fe+1.5%Cr+0.2%Mo+0.8%C	CrL+0.8%C	Astaloy CrL	1.5	0.2	0.8	Bal.
Fe+ 3%Cr+0.5%Mo+0.8%C	CrM+0.8%C	Astaloy CrM	3	0.5	0.8	Bal.

The mixed powders were then compacted in a steel die with zinc stearate lubricated walls into ISO 2740 dog-bone tensile test bars. Isothermal sintering was carried out in dry (10 ppm moisture) $5\%H_2 - 95\%N_2$ atmosphere in a laboratory horizontal tube furnace at 1120 and 1250°C for 60 minutes, employing convective (65°C min^{-1}) cooling. The heating and cooling rates were 75°C/min and 60°C/min, respectively. After sintering, the sintered steels were tempered at 200°C for 60 minutes in air. The green (d_1) and as-tempered (d_2) densities of the compacts are summarised in Tables 2-3.

To determine the effect of processing parameters on the structure and mechanical properties of sintered steels, the densities (both green and as-sintered as tempered), transverse rupture strength (TRS), ultimate tensile strength (UTS), elongation, $R_{0.2}$ yield offset and cross-section microhardness (Fig. 1) were measured.

Tensile tests were carried out on a MTS testing machine with a cross-head speed 1 mm/min. Parameters of tensile test were defined in accordance with PN-EN 10002-1 standard. Bend tests were performed on a ZD10 testing machine, with the tensile surface being that pressed by the upper punch. Hardness testing, at 0.5 mm intervals, was made on a Innovatest microhardness tester with a 100 G load (Fig. 1).

TABLE 2

Density of green compacts, d_1, and sintered steels, d_2 – mean values (10 samples) – geometrical method

Designation	1120°C			1250°C		
	Mean density, g/cm^3		Density changes, %	Mean density, g/cm^3		Density changes, %
	d_1	d_2		d_1	d_2	
CrA+0.4%C	6.67±0.02	6.68±0.02	0.15	6.70±0.02	6.70±0.01	0.00
CrL+0.4%C	6.76±0.03	6.79±0.03	0.44	6.71±0.02	6.74±0.01	0.45
CrM+0.4%C	6.65±0.02	6.65±0.02	0.00	6.67±0.01	6.68±0.02	0.15
CrA+0.8%C	6.65±0.02	6.70±0.03	0.75	6.74±0.02	6.75±0.01	0.15
CrL+0.8%C	6.77±0.02	6.67±0.03	1.48	6.77±0.01	6.77±0.01	0.00
CrM+0.8%C	6.70±0.02	6.69±0.02	-0.15	6.70±0.02	6.71±0.02	0.15

TABLE 3

As-sintered density, d_2, defined by geometrical and Archimedes methods – mean values (10 samples)

Designation	1120°C			1250°C		
	d_2, g/cm^3		Density changes, %	d_2, g/cm^3		Density changes, %
	Geometrical	Archimedes		Geometrical	Archimedes	
CrA+0.4%C	6.68±0.02	6.91	3.44	6.70±0.01	7.01	4.69
CrL+0.4%C	6.79±0.03	6.79	0.00	6.74±0.01	6.90	2.37
CrM+0.4%C	6.65±0.02	6.91	3.91	6.68±0.02	7.00	4.79
CrA+0.8%C	6.70±0.03	6.94	3.58	6.75±0.01	7.03	4.15
CrL+0.8%C	6.67±0.03	6.70	0.45	6.77±0.01	7.14	5.47
CrM+0.8%C	6.69±0.02	6.80	1.64	6.71±0.02	7.06	5.22

TABLE 4

Mechanical properties of the PM steels – mean values (10 samples)

Designation	1120°C					1250°C				
	UTS, MPa	$R_{0.2}$, MPa	A, %	TRS, MPa	HV 0.01	UTS, MPa	$R_{0.2}$, MPa	A, %	TRS, MPa	HV 0.01
CrA+0.4%C	406±22	234±20	4.30±0.65	823±133	177±30	457±22	333±14	6.55±0.92	942±148	137±20
CrA+0.8%C	605±35	361±23	3.17±0.55	1364±68	327±66	725±30	459±33	5.70±0.57	1362±370	223±54
CrL+0.4%C	429±36	244±13	3.94±0.87	971±133	243±64	465±33	321±27	4.87±1.12	1066±185	169±38
CrL+0.8%C	457±30	331±27	1.32±0.26	930±75	327±90	742±28	387±17	4.16±0.42	1470±320	229±55
CrM+0.4%C	629±63	413±20	2.07±0.40	1383±155	145±33	770±35	422±28	4.84±0.42	1249±290	256±50
CrM+0.8%C	795±54	522±24	2.36±0.26	1780±129	175±60	964±26	469±22	5.53±0.34	1415±135	401±113

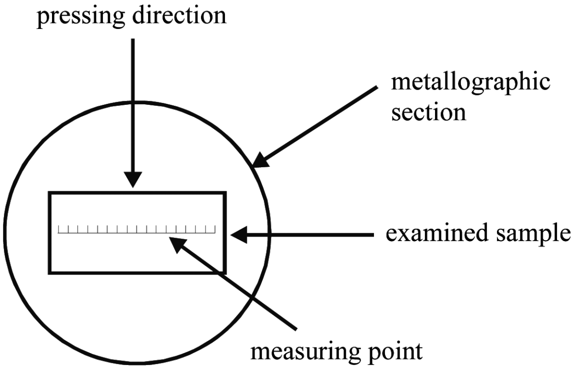

Fig. 1. The scheme of microhardness testing; the distance between test points – 0.5 mm

Metallographic investigations were carried out on 3% Nital etched samples, prepared according to the procedure described in [30] on a Leica DM4000M optical microscope at a magnification of 500x. Fractographic examinations were made on a Hitachi S-3500N scanning transmission electron microscope (SEM), equipped with EDS (made by Noran).

3. Results and discussion

Tables 2-5 present the results of physical and mechanical investigations of Fe-(Cr)-(Mo)-C and Fe-(Cr)-C PM steels. In Table 2 mean values of densities of green compacts and as-sintered / as-tempered steels are presented. Independently of powder mixture chemical composition, the green densities of compacts, geometrically measured, were in the range of 6.65-6.77 g/cm^3. Both the sintering temperatures had negligible effects on the density of the steels. After sintering at 1120°C the highest swelling was observed for CrL+0.4C (+0.44%) and the highest shrinkage was recorded for CrL+0.8C (-1.48%). After sintering at 1250°C small swelling (approx. 0.15%-0.45%) was observed.

The Archimedes method of determining density of sintered steels is much more precise, Table 3. As-sintered densities of steels varied from 6.70 g/cm^3 to 6.94 g/cm^3 and from 6.90 g/cm^3 to 7.14 g/cm^3 for steels sintered at 1120 and 1250°C, respectively. The results show an increase of as-sintered / as-tempered density with increasing sintering temperature. The data presented in Tables 2 and 3 indicate that sintering at the lower temperature limits the rate of diffusion processes taking place in the being sintered material.

Mechanical properties of the steels are presented in Table 4. Using formula (1), the percentage changes in mechanical properties with increase of sintering temperature are summarised in Table 5.

$$\Delta P = \frac{P_{1250^\circ} * 100}{P_{1120^\circ C}} - 100 \qquad (1)$$

where: ΔP – property changes, $P_{1120^\circ C}$, $P_{1250^\circ C}$ – property recorded after sintering at 1120 or 1250°C, respectively.

Mechanical properties also depend on the chemical composition of the base pre-alloyed iron powder. With increasing amount of alloying elements, mechanical properties strongly increased. The highest UTS and TRS after sintering at 1120°C were recorded for CrM+0.8%C (795 MPa and 1780 MPa, respectively). For steels sintered at 1250°C the highest UTS was recorded for CrM+0.8%C steels (964 MPa) and TRS – for CrL+0.8%C – 1470 MPa. It can be pointed out the mechanical properties of CrA+0.8C are comparable or even better than those recorded for CrL+0.8%C (Table 4).

TABLE 5

Property changes after sintering at 1250°C

Designation	ΔR_m, %	$\Delta R_{0.2,}$ %	ΔA, %	ΔR_g, %	ΔHV, %
CrA+0.4%C	13	42	52	14	-23
CrA+0.8%C	20	27	80	0	-32
CrL+0.4%C	8	32	24	10	-30
CrL+0.8%C	62	17	215	58	-30
CrM+0.4%C	22	2	134	–10	77
CrM+0.8%C	21	–10	134	–21	129

Increasing the carbon content, irrespective of the sintering temperature, increased the strength properties, Table 4. On the other hand, plastic properties of theses steels decreased with increasing carbon content. This effect was observed for all chemical compositions investigated.

Because of the high price of Ni and its cancerogenic effect, the Ni-containing steels can be substituted, with success, by (Mn)-(Cr)-(Mo) PM steels reaching a comparable or higher mechanical properties. On the other hand, the properties of the investigated steels are lower than those given in Table 6: values of mechanical properties for Astaloy CrA, Astaloy CrL and Astaloy CrM-based steels reported in Refs. 31, 32.

TABLE 6

The properties of PM steels based on Astaloy CrA, Astaloy CrL and Astaloy CrM pre-alloyed iron powders [31, 32]

Alloy	Sintering temperature, °C	$R_{0,2}$ offset, MPa	UTS MPa	HV 10	KC, J/cm^2 (Impact energy, J)	A, %
CrA+0.8C	1120	~ 570	>700	Not defined	(20)	1.2
	1250	~ 700	~ 900	Not defined	(30)	2.4
CrL+0.4C	1120	420	560	180	17	7-8
	1250	600	850	250	32	1.6
CrL+0.8C	1120	560	700	210	10-12	~1
	1250	700	950	200	28	2.5
CrM+0.4C	1120	625	800	250	20	1.5
	1250	850	1100	325	28	2.5
CrM+0.8C	1120	750	1000	450	15	0.25
	1250	800	1050	340	22	1.5

The presence of carbon in the investigated steels, in amount of 0.8%, increased their hardness; it was higher and toughness was lower than those in low-carbon steels. The higher carbon content in the investigated steels does not improve their tensile and bend strengths. Even if a high strength microstructure of the matrix was obtained, the problem was in interfaces contaminated with oxide phases which results in low impact properties, but this is problem of admixed manganese.

Characteristic microstructures and fractographs obtained for the tested alloys at both temperature are shown in Figs. 2-3 and 4-5, respectively. All the microstructures examined were complex and heterogeneous. On the basis the data presented in Ref. 33, in Table 7 characteristic structural constituents

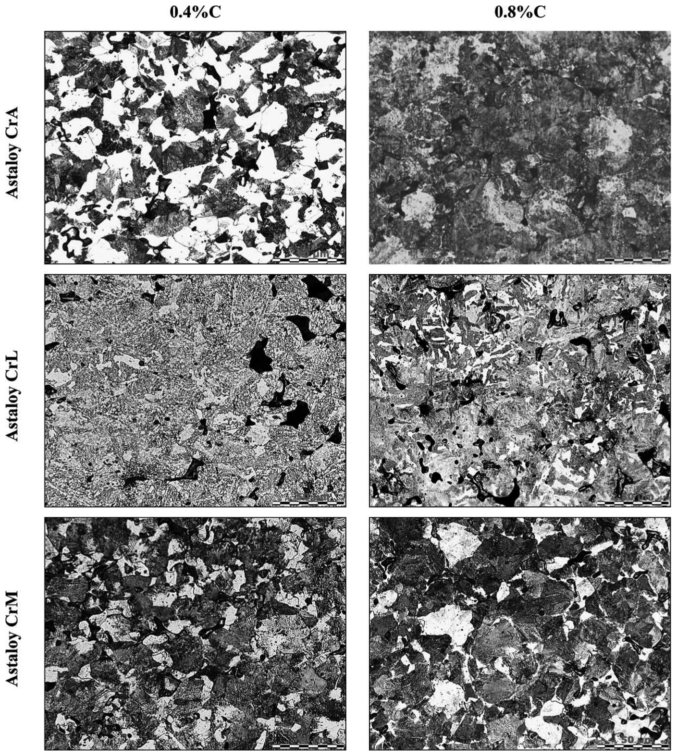

Fig. 2. The microstructures of steels sintered at 1120°C (marker 50 μm)

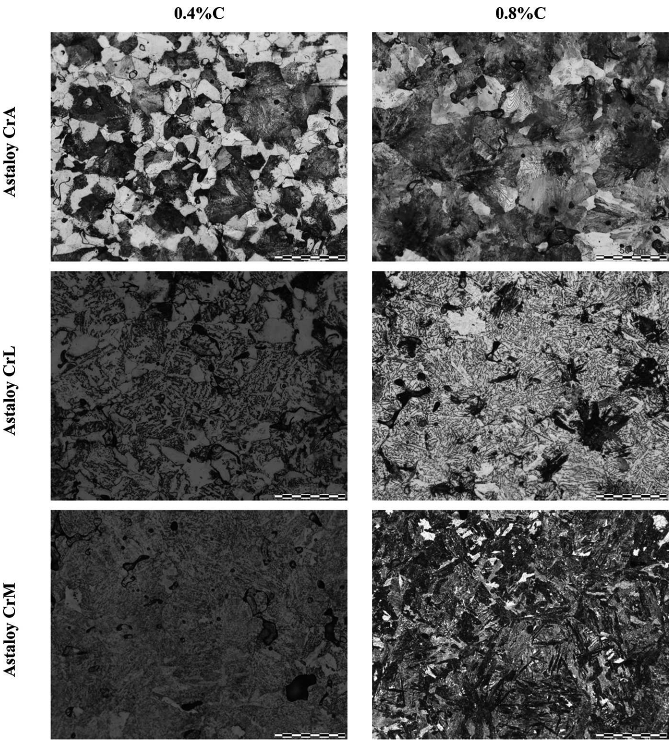

Fig. 3. The microstructures of steels sintered at 1250°C (marker 50 μm)

TABLE 7

Characteristic structural constituents of investigated steels after sintering at 1120 and 1250°C; F – ferrite, P – pearlite, B – bainite, M – martensite

Designation	Sintering temperature, °C	
	1120	1250
CrA+0.4%C	F + P	F+P
CrA+0.8%C	P + F	P
CrL+0.4%C	F + P + B	F + B
CrL+0.8%C	B + P + M	B + M
CrM+0.4%C	B + M	B + M
CrM+0.8%C	M + B	M + B

of investigated steels after sintering at 1120 and 1250°C are presented. The microstrucutre of investigated steels containing 0.3%C sintered at 1120°C consists of: ferrite + pearlite (CrA+0.4%C and CrA+0.8%C), ferrite + pearlite + bainite (CrL+0.4%C), bainite + pearlite + martensite (CrL+0.8%C) and bainite + martensite (CrM+0.4%C and Cr+0.8%C). The results presented in Table 4 correspond well with the microstructure (Figs. 2-3) and the fracture of the investigated steels (Figs. 4-5).

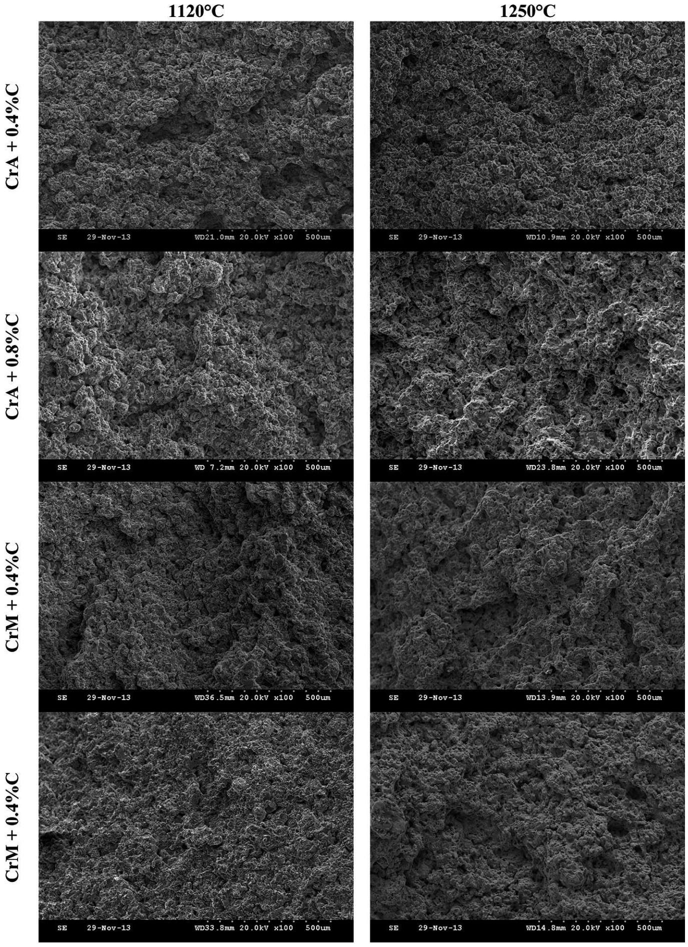

Fig. 4. The fracture surfaces of PM steels based on Astaloy CrA and Astaloy CrM pre-alloyed powder (SEM)

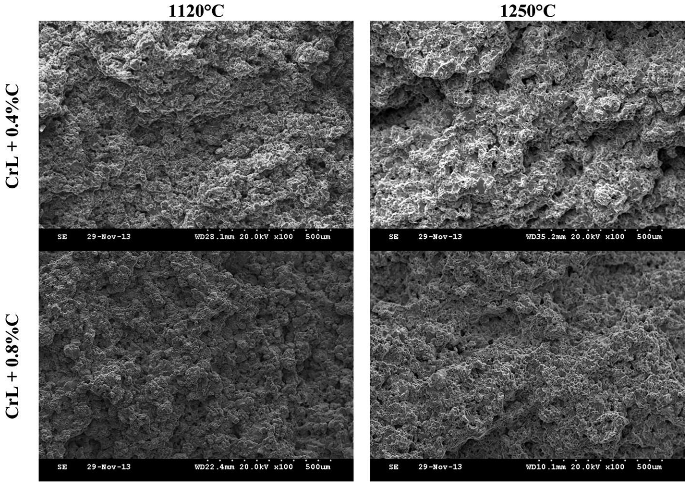

Fig. 5. The fracture surfaces of PM steels based on Astaloy CrL pre-alloyed powder (SEM)

A high-carbon content results in the complex and high-strength sintered microstructure containing martensite and bainite (CrM+0.8%C and CrL+0.8%C steels). For Astaloy CrA-based steels with addition of 0.8%C, ferrite + pearlite or pearlite (after sintering at 1120 and 1250°C, respectively) was observed. These results are in a good agreement with those presented in Ref. 32.

The fracture surface morphology after tensile tests (Figs. 4 and 5) is strongly dependent on the type of microstructure, which is controlled by carbon content and sintering temperature. For alloys based on both Astaloy CrL and Astaloy CrM powders with lower carbon content, it is dominated by interparticle failure with shallow dimples, some small cleavage facets and intergranular decohesion. In some areas of ductile dimple failure, particularly for high temperature sintering, local plastic deformation occurred, which corresponds with achieved values of plasticity, expressed by tensile strains of more than 5%.

The failure is controlled by quality of interface areas, particularly of bainite packet surfaces and by cleavage in martensite. The result is a mixed character of the fracture surface, which consists of shallow dimples and cleavage facets. Both lower sintering temperature and lower chromium content lead to higher amount of intergranular decohesion (Fig. 4), which is connected with the formation of carbide phases at grain boundaries. In the case of carbon content of 0.8% and higher sintering temperature the amount of cleavage facets increases.

4. Conclusions

The investigations allowed the following conclusions to be drawn:

1. The investigated steels correspond to medium strength steels which are used for structural parts in ferrous powder metallurgy and with success can substitute traditional, expensive PM steels.
2. Higher temperature resulted in a significant increase in mechanical and plastic properties for all alloy variants.
3. Increasing the carbon content resulted in an increase of mechanical properties.
4. Sintered steels based on Astaloy CrM prealloyed iron powder showed the best mechanical properties, independently of carbon concentration in the steels.
5. In alloys based on Astaloy CrM sintered at the higher temperature, bainite and martensite were found, which caused a reduction of mechanical properties and increase of hardness.
6. Ferritic/pearlitic or pearlitic sintered steels based on Astaloy CrA prealloyed iron powder seems to be a good alternative to the other investigated steels.

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