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THE MICROSTRUCTURE AND PROPERTIES OF LOW-CARBON PM Mn-Cr-Mo STEELS SINTERED UNDER DIFFERENT CONDITIONS

MIKROSTRUKTURA I WŁASNOŚCI MECHANICZNE NISKOWĘGLOWYCH, SPIEKANYCH STALI Mn-Cr-Mo WYTWARZANYCH W RÓŻNYCH WARUNKACH

The paper presents the effect of sintering conditions on the microstructure and mechanical properties of low-carbon Mn-Cr-Mo PM steels. It was proved there is no effect of tempering temperature on the properties of Astaloy CrL-base steels, sintered at 1250°C in 5%H_2-95%N_2 mixture as compared with the properties of those sintered at 1120°C. The properties of Astaloy CrM-based steels, sintered at 1250°C in air were comparable or higher to Astaloy CrL-based steels. The addition of lump of ferromanganese was not sufficient for metal oxides reduction. The structure investigation confirmed the earlier observations that Mn-Cr-Mo PM steels have predominantly martensitic or martensitic/bainitic microstructure.

Keywords: structural PM materials, PM Mn steels, sintering process, sintering atmosphere, microstructure, mechanical properties

W artykule przedstawiono wpływ parametrów spiekania na mikrostrukturę i własności mechaniczne spiekanych, niskowęglowych stali manganowo-chromowo-molibdenowych. W pracy wykazano, że własności mechaniczne stali wykonanej na bazie proszku stopowego Astaloy CrL (1,5%Cr, 0,2%Mo, reszta Fe) spiekanej w temperaturze 1250°C w atmosferze 5%H_2-95%N_2 nie zależą od temperatury odpuszczania, czego nie stwierdzono podczas badań własności wytrzymałościowych tych stali wytwarzanych w niższej temperaturze spiekania (1120°C). Ponadto, prowadzone badania wykazały, że stale wytworzone na bazie proszku stopowego Astaloy CrM (3%Cr, 0,5%Mo, reszta Fe) po spiekaniu w temperaturze 1250°C w atmosferze powietrza charakteryzowały się porównywalnymi lub wyższymi własnościami mechanicznymi niż stale wykonane na bazie proszku stopowego Astaloy CrL, wytwarzane w tych samych warunkach. Podczas spiekania w powietrzu zastosowano odłamkowe cząstki żelazomanganu o łącznej masie 52 g, które umieszczano w bezpośrednim sąsiedztwie próbek (całkowita masa próbek 490 g) w stalowej łódce, w celu redukcji tlenków. Jak wykazały badania, dodatek żelazomanganu był niewystarczający, aby zapewnić kompletną redukcję tlenków.

Badania metalograficzne spiekanych stali manganowo-chromowo-molibdenowe wykazały, że głównym składnikiem strukturalnych tych stali, niezależnie od temperatury spiekania, był martenzyt lub mieszanina martenzytu i bainitu.

1. Introduction

Manganese and chromium are two important elements in steels, which show effective strengthening. The issue of sintering low alloy steels containing these metals having high affinity for oxygen is one of the most important topics in powder metallurgy (PM), as testified by the number papers on this subject that have taken place over the last few years [1-11]. The question of the role of "micro-atmosphere" in the development of microstructure is fundamental for understanding of sintering process, especially the relationship between microstructure to mechanical properties evolution. The thermodynamics bonds concerning carbon equilibrium during sintering were discussed and possible interactions between steel and various controlled atmosphere were examined [12-15]. Therefore it is anticipated that this topic would generate a great deal of interest among powder metallurgy specialists.

The PM industry needs economical ways of producing components with higher densities in order to effect the stepwise improvement in mechanical properties necessary to compete with highly loaded wrought and machined components. To research a potentially economical route for production of components made of Fe-Mn-Cr-Mo-C structural steels, the ways of maximising mechanical properties were investigated. The slow cooling of large masses in semi-closed containers in the sintering furnace favours bainite formation because of high hardenability of Mn Cr, Mo alloyed steels. Correct sinter-hardening of these steels should lead to a tough bainitic core with a high wear resistant surface. The new processing conditions aim to make use of carbothermic reduction of metal oxides by promotion of a local "micro-climate" or "micro-atmosphere" around the sintered components, with low oxygen potential and high CO/CO_2 ratio. It is well known, from iron and steel-making processes, that at temperatures

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>900°C carbon monoxide is a more efficient reducing gas than pure dry hydrogen. Also CO_2 is efficiently reduced to CO by solid carbon by means of the Boudouard reaction at temperatures >927°C. It was proposed to model theoretically the metal-metal oxide-carbon reactions to predict the generation of CO and CO_2 versus temperature and the projected efficiency for gaseous-metal oxide reduction. These analyses were used to corroborate the theoretical modelling and also to help with design improvements of semi-closed container systems [1, 14, 16]. The relatively small volume of reducing gas (CO/CO_2) in these containers ensures that the process is more eco-friendly than when either flowing endogas (unacceptable due to its wetness) or cracked ammonia (or other nitrogen-hydrogen, minimum 10% mixture) plus methane addition is employed, as currently in industry. The use of carbon and/or manganese vapour producing getter systems will be investigated for the purposes of increased CO/CO_2 generation and drying of the "micro-climate", thus ensuring low oxygen potential and best possible reducing conditions. Avoidance of formation of deleterious manganese and chromium oxides and chromium nitrides has already been demonstrated [14, 16].

Various semi-closed container designs have been used previously during authors work [17-22], in order to understand and develop of sintering parameters for Mn and Cr containing PM alloys.

This paper aims to determine the processing conditions necessary for standard and high temperature sintering in nitrogen rich, non-flammable atmospheres of PM ferrous structural parts, which contain the easily oxidisable chromium and manganese in addition to carbon and molybdenum. These components are to possess high fatigue strength, good dimensional accuracy, be fully recyclable and of lower cost than equivalent wrought and machined parts. Recyclability is achieved by substitution of the common (for PM) alloying elements of copper and nickel by chromium and manganese. Recyclable components are of paramount importance to automotive manufacturers due to increasingly stringent legislation that now sees the manufacturers responsible for the "whole life cycle" of vehicles, i.e. "birth to grave". Another element often used in PM is the expensive and potentially carcinogenic nickel. Its exclusion as an alloying element can only make for a safer workplace. Also the issue has a high profile in public awareness because of a series of magazine articles.

2. Experimental procedure

The commercial, pre-alloyed Höganäs Astaloy CrL and Astaloy CrM powders were used as the base materials. 3 mass % of manganese, in the form of low-carbon (1.3% C) ferromanganese (77% Mn) powder (with particle size below 20μm), and 0.15 mass % of ultra fine graphite powder grade C-UF were added to the base powders in order to prepare two mixtures based on Astaloy CrL and Astaloy CrM, respectively (Fig. 1):

- Fe-3%Mn-1.5%Cr-0.2%Mo-0.15%C,
- Fe-3%Mn-3%Cr-0.5%Mo-0.15%C.

The powders were mixed in a Turbula mixer for 30 min, and compacted in steel dies with zinc stearate lubricated walls. Two types of compacts were prepared: 55×10×5 mm TRS specimens and ISO 2740 dog-bone tensile test bars. 40 compacts of each type were prepared from both mixtures.

Sintering was carried out in the laboratory horizontal tube furnace at 1120°C and 1250°C for 60 minutes. The heating and cooling rates were 75°Cmin^{-1} and 65°Cmin^{-1}, respectively. The sintering atmospheres were dry (10 ppm moisture) 5% H_2-95% N_2 atmosphere and air (Table 1). The flow of gas mixture was very slow, approx. 1 ml min^{-1}, to get stable conditions inside the boat. Sintering in air was carried out with the presence of Mn vapours coming from a lumps of the ferromanganese placed in the semi-closed stainless steel container. The mass of lump of FeMn was 52 g and it was added per 490 g of compacts, both rectangular and 10 ISO 2740. The total number of compacts during single sintering process was 20 – 10 rectangular and 10 ISO 2740. A half of sintered number of each type of samples were tempered at 200°C for 60 minutes in air.

TABLE 1

The scheme of sintering conditions

Astaloy CrL + 3%Mn + 0.15%C			
Sample No		Temperature, °C and sintering atmosphere	
Not tempered	Tempered at 200°C	1120	1250
CrL-A	CrL-B	-	$5H_2$-$95N_2$
CrL-C	CrL-D	-	air + FeMn
CrL-E	CrL-F	$5H_2$-$95N_2$	-
CrL-G	CrL-H	air + FeMn	-
Astaloy CrM + 3%Mn + 0.15%C			
Sample No		Temperature, °C and sintering atmosphere	
Not tempered	Tempered at 200°C	1120°C	1250°C
CrM-A	CrM-B	-	$5H_2$-$95N_2$
CrM-C	CrM-D	-	air+ FeMn
CrM-E	CrM-F	$5H_2$-$95N_2$	-
CrM-G	CrM-H	air + FeMn	-

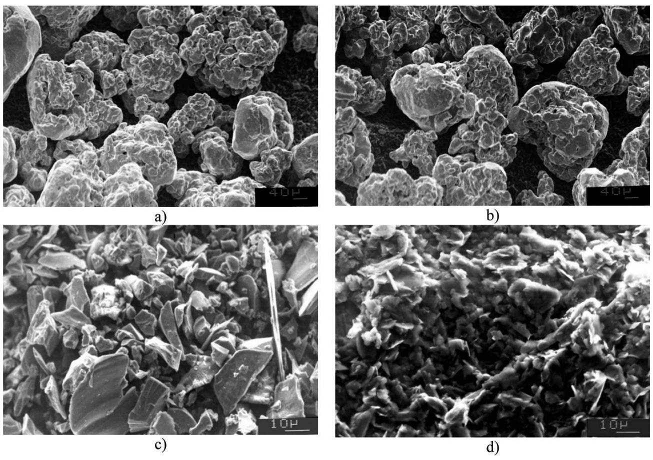


Fig. 1. Micrographs of base powders: a) pre-alloyed Astaloy CrL powder, b) pre-alloyed Astaloy CrM powder, c) ferromanganese Elkem powder, d) graphite powder grade C-UF

The green densities of compacts were established geometrically while the sintered densities were measured by Archimedes method. The green and as-sintered densities of compacts, d_0 and d_1, respectively, are summarised in Table 2.

TABLE 2
Green densities, d_0, and as-sintered densities, d_1, of Fe-Mn-Cr-Mo-C PM steels – mean values for 40 (green compacts) and 20 (as-sintered materials) measurements

Green compacts	Astaloy CrL + 3%Mn + 0.15%C		Astaloy CrM + 3%Mn + 0.15%C	
	Rectangular	ISO 2740	Rectangular	ISO 2740
	d_0, g/cm^3	d_0, g/cm^3	d_0, g/cm^3	d_0, g/cm^3
	6.82±0.12	6.63±0.02	6.65±0.07	6.55±0.06
Compacts sintered at:	d_1, g/cm^3	d_1, g/cm^3	d_1, g/cm^3	d_1, g/cm^3
1120°C	6.83±0.14	6.62±0.02	6.65±0,19	6.51±0,04
1250°C	6.84±0.15	6.62±0.02	6.77±0,06	6.59±0,02

Sintered compacts were mechanically tested. In the frame of mechanical testing, bending (TRS) and tensile (UTS) strength tests as well as toughness and hardness were measured. Metallography investigation of sintered Mn-Cr-Mo steels was carried out using LOM technique.

3. Results

The mechanical properties of investigated PM steels are summarised in Tables 3-4 and in Figures 2-9. Leco instruments were employed to check the carbon, oxygen and nitrogen contents in Fe-Mn-Cr-Mo-C PM steels.

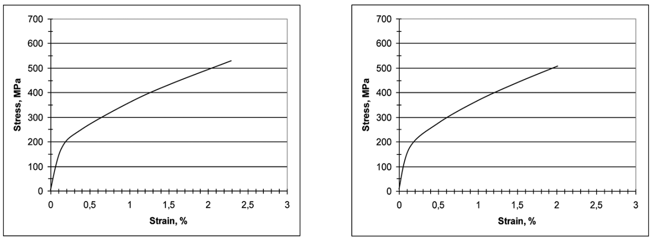
Following the results of presented in Table 5, higher sintering temperature contributes the decarburization effect in investigated steels. This effect is connected with carbothermic reaction between C and O_2 which can be possible during the whole heating and sintering steps. This phenomenon was widely reported by Cias et al [16]. Also the lower oxygen content in PM steel after sintering at 1250°C suggests the carbon–oxygen reactions. It has to be also pointed out that sintering in air with additions of FeMn, irrespective of sintering temperature, contributes to decreasing decarburization effect due to the shortage of hydrogen. Higher nitrogen level in investigated steels can be explained by nitrogen-rich atmosphere; also porosity which is seen in Figs. 10-11, play important role in nitriding Mn-Cr-Mo PM steels.

TABLE 3
Mechanical properties of Fe-3Mn-Cr-Mo-C steels based on Astaloy CrL pre-alloyed powder mean values and standard deviations

Astaloy CrL + 3%Mn + 0.15%C						
Description	UTS, MPa	Strain-to-failure, %	$R_{0.2}$.yield offset, MPa	TRS, MPa	Toughness, J/cm^2	HV 30
CrL-A	593±53	2.57±0.40	543±28	1104±218	6.19±1.01	231±34
CrL-B	592±53	2.54±0.22	540±51	1114±190	5.89±0.84	159±25
CrL-C	580±58	2.79±0.55	506±70	1171±151	5.85±1.33	243±37
CrL-D	505±154	2.19±0.88	483±57	1158±115	7.10±1.12	224±21
CrL-E	508±19	2.16±0.14	430±57	969±140	4.66±0.72	218±15
CrL-F	506±59	2.05±0.35	432±26	979±152	5.08±0.86	170±40
CrL-G	513±58	2.15±0.34	426±52	1088±109	4.39±0.76	185±59
CrL-H	603±42	2.57±0.38	468±34	1138±204	5.26±0.86	202±35

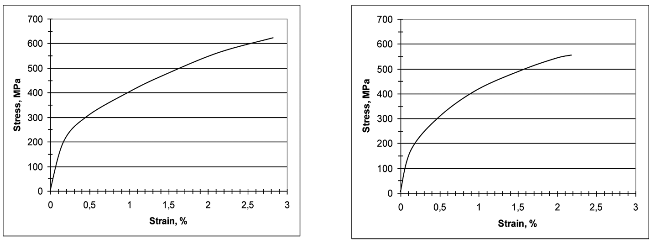
TABLE 4
Mechanical properties of Fe-3Mn-Cr-Mo-C steels based on Astaloy CrM pre-alloyed powder mean values and standard deviations

Astaloy CrM + 3%Mn + 0.15%C						
Description	UTS, MPa	Strain-to-Failure,%	$R_{0.2}$.yield offset, MPa	TRS, MPa	Toughness, J/cm^2	HV 30
CrM-A	689±62	1.30±0.23	668±54	1083±44	4.92±1.35	247±30
CrM-B	739±47	1.38±0.15	698±49	1178±32	5.66±0.75	256±36
CrM-C	619±66	2.27±0.53	502±63	1191±96	6.12±1.27	272±53
CrM-D	642±38	2.08±0.26	561±51	1203±120	5.35±1.23	300±35
CrM-E	431±62	1.28±0.30	421±76	897±83	4.45±0.54	246±66
CrM-F	552±65	1.63±0.27	536±40	976±135	3.32±0.66	191±60
CrM-G	514±75	1.78±0.24	433±51	906±80	3.55±0.58	184±71
CrM-H	541±79	1.59±0.35	554±26	809±120	3.61±0.43	300±20



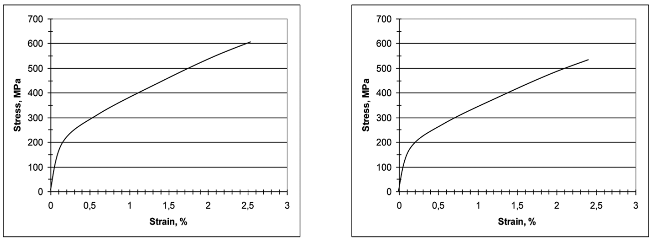
a) b)

Fig. 2. Representative tensile curves for Astaloy CrL-based 3%Mn + 0.15%C PM steels sintered at 1120°C in 5%H_2-95%N_2 mixture; a) not tempered and b) after tempering



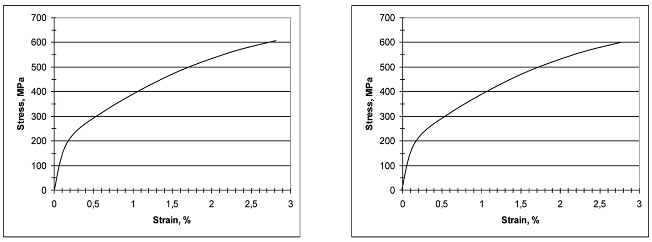
a) b)

Fig. 3. Representative tensile curves for Astaloy CrL-based 3%Mn + 0.15%C PM steels sintered at 1120°C in air + FeMn (added in the form of a lump); a) not tempered and b) after tempering



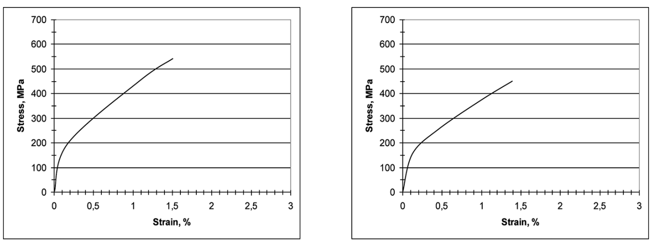
a) b)

Fig. 4. Representative tensile curves for Astaloy CrL-based 3%Mn + 0.15%C PM steels sintered at 1250°C in 5%H_2-95%N_2 mixture; a) not tempered and b) after tempering



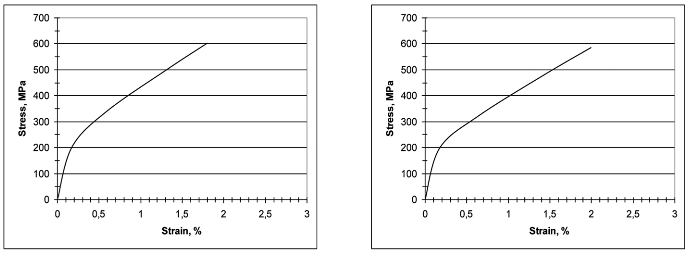
a) b)

Fig. 5. Representative tensile curves for Astaloy CrL-based 3%Mn + 0.15%C PM steels sintered at 1250°C in air + FeMn (added in the form of a lump); a) not tempered and b) after tempering



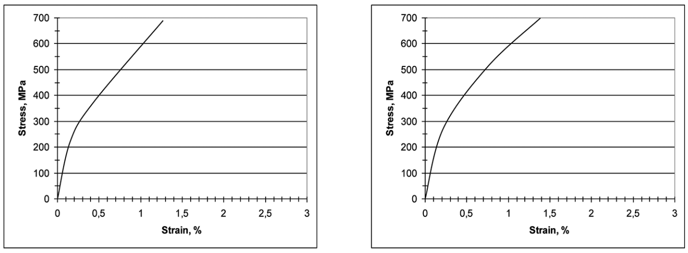
a) b)

Fig. 6. Representative tensile curves for Astaloy CrM-based 3%Mn + 0.15%C PM steels sintered at 1120°C in 5%H_2-95%N_2 mixture; a) not tempered and b) after tempering



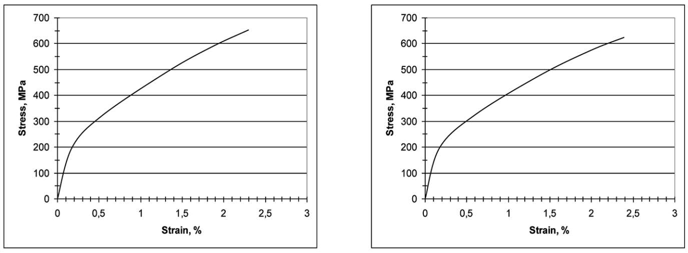
a) b)

Fig. 7. Representative tensile curves for Astaloy CrM-based 3%Mn + 0.15%C PM steels sintered at 1120°C in air + FeMn (added in the form of a lump); a) not tempered and b) after tempering



a) b)

Fig. 8. Representative tensile curves for Astaloy CrM-based 3%Mn + 0.15%C PM steels sintered at 1250°C in 5%H_2-95%N_2 mixture; a) not tempered and b) after tempering



a) b)

Fig. 9. Representative tensile curves for Astaloy CrM-based 3%Mn + 0.15%C PM steels sintered at 1250°C in air + FeMn (added in the form of a lump); a) not tempered and b) after tempering

TABLE 5

Chemical composition of investigated not tempered 3%Mn-(Cr)-(Mo)-0.15%C PM steels

Steel description	Sintering temperature, °C / atmosphere	Chemical composition		
		O_2, %	N, %	C, %
Astaloy CrL + 3%Mn + 0.15%C	1250 / 5%H_2-95%N_2	0.305	0.0664	0.154
	1250 / air + FeMn	0.321	0.0500	0.182
	1120 / 5%H_2-95%N_2	0.284	0.0524	0.174
	1120 / air + FeMn	0.300	0.0476	0.187
Astaloy CrM + 3%Mn + 0.15%C	1250 / air + FeMn	0.275	0.0664	0.164
	1120 / 5%H_2-95%N_2	0.327	0.0794	0.183
	1120 / air + FeMn	0.348	0.0732	0.172

The heterogeneous microstructure of investigated PM steels observed in bright field (Figs. 10 and 11) mainly consists of martensite or martensite+bainite (lower and upper); also a lot of upper bainitic islands, homogeneous arranged, were observed.

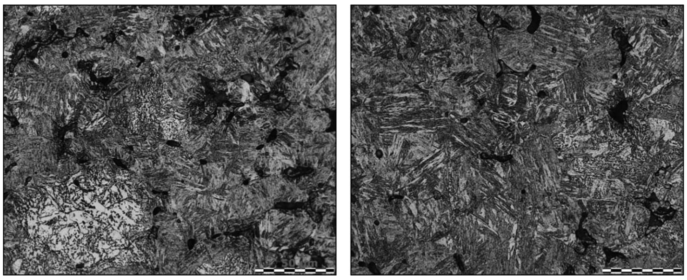


Fig. 10. The microstructure of Astaloy CrL-based not tempered steel sintered at 1120°C (left) and 1250°C (right)

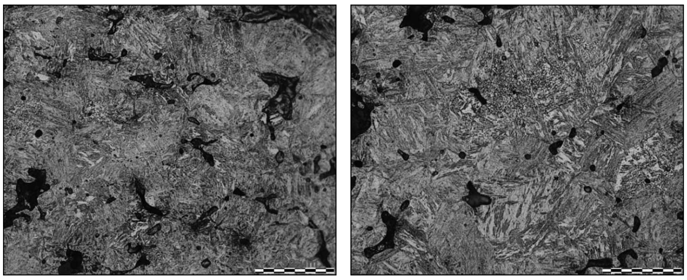


Fig. 11. The microstructure of Astaloy CrM-based not tempered steel sintered at 1120°C (left) and 1250°C (right)

4. Discussion

The strength properties of PM Mn-Cr-Mo-C steels obtained during investigations indicate that these steels can be classified as medium-to-high strength wrought steels.

As was shown in Tables 2, compacts based on Astaloy CrL pre-alloyed powder are characterised by a little bit higher densities than those based on Astaloy CrM powder. It can be connected with better compressibility of powder mixture containing less chromium and molybdenum.

Mechanical properties of investigated PM steels were summarised in Tables 3-4 and in Figures 2-9. High temperature sintering of steels based on Astaloy CrL powder in 5%H_2-95%N_2 mixture and their tempering does not influence on strength properties of investigated steels. UTS and TRS strengths and $R_{0,2}$ yield offset are comparable for tempered and not tempered samples; toughness and hardness values recorded for not tempered samples increased by 4.8% and 25% than those obtained for as-tempered steels.

There are some materials e.g. gray cast iron or porous steels for which the initial elastic portion of the stress-strain curve is not linear; hence, it is not possible to determine a modulus of elasticity as for wrought steel. For this nonlinear behaviour, either tangent or secant modulus is used. Tangent modulus is taken as the slope of the stress-strain curve at some specified level of stress, while secant modulus represents the slope of a secant drawn from the origin to some given point of the stress-strain curve. For the investigated specimens the proportional limit stress was ~160MPa and Young modulus at this limit (measured either as secant or tangent modulus) was 150GPa (146-160GPa measured using ultrasonic technique). The Young's modulus of PM steels, based on Astaloy CrL and Astaloy CrM pre-alloyed powders, containing 0.3% of carbon, evaluated by the supersonic method along the sample, was in the range of 144-170 GPa [23].

The mechanical properties of both group of steels (Astaloy CrL and Astaloy CrM-based materials) are comparable, irrespective of sintering atmosphere. Not tempered steels, sintered in air with addition of 52g of FeMn obtained higher mechanical properties. After sintering at 1120°C in 5%H_2-95% N_2 mixture, irrespective of heat treatment, the comparable mechanical properties of investigated PM steels were obtained. Mechanical properties of steels based on Astaloy CrM pre-alloyed powder, sintered at 1250°C in air with addition of 52g of FeMn were higher or comparable to the properties of low-chromium, low-molybdenum steels sintered at the same temperature in 5%H_2-95% N_2 atmosphere. A specific characteristic of manganese in relation to sintering mechanisms is its vapour pressure, the highest of all the alloying elements in PM structural steels. Its significance was first recognised by Salak [22, 24], who reported that manganese sublimation and evaporation plays a significant part in such phenomena as homogenisation and self-gettering action of Mn vapour. The observed rapid alloying of iron particles in Mn steels can only be accounted by transport of manganese *via* gaseous phase. High manganese vapour pressure make possible manganizing of the sintered alloy, a process the diffusion of manganese into the surface of a metal, particularly the steel compacted powder particles, and improve its mechanical properties. This may be achieved by sintering the compacts (open-porous material) at 1100-1250°C in a sealed boat packed with compacts and ferromanganese lumps and with an inert gas/manganese vapour atmosphere. Additionally manganese and carbon loss is lowered.

The effect of low sintering at 1120°C followed by tempering at 200°C on increasing the strength properties, irrespective of chemical composition of sintering atmosphere, in Astaloy CrL based steel was observed. When tempering wasn't carried out, the higher properties were recorded for steel sintered in air with addition of 52 g of FeMn.

The results of chemical analysis presented in Table 5 has showed, that reduction of oxides is more advanced in 5%H_2-95% N_2 atmosphere. It can be pointed out that the addition of 52 grams of ferromanganese is not sufficient to oxide reduction presented in sintered steels.

Higher amount of nitrogen in sintered compacts can be explained by nitriding in nitrogen-rich atmosphere. The highest decarburization was observed for Astaloy CrL-based steels sintered at 1250°C in the presence of hydrogen. This phenomenon was also recorded in Astaloy CrM-based steels sintered in air with addition of 52g FeMn.

5. Conclusions

Assuming the present work, the following conclusions can be drawn:

1. The effect of heat treatment on the mechanical properties of Astaloy CrL-based steels sintered at 1250°C in 5%H_2-95%N_2 mixture was not observed.
2. Mechanical properties of Astaloy CrM-based steels sintered at 1250°C in air + Mn vapour were comparable or higher than those recorded for low-chromium, low-molybdenum PM steels.

3. Low temperature sintering at 1120°C followed by tempering at 200°C, irrespective of sintering atmosphere, allow increasing strength properties of low-chromium, low-molybdenum PM steels.
4. Not tempered, low-chromium, low-molybdenum PM steels sintered in air with addition of 52g FeMn obtained higher mechanical properties than those sintered in nitrogen/hydrogen mixture.
5. The addition of lump of ferromanganese in amount of 52 grams is not sufficient for oxides reduction.
6. Both higher sintering temperature and the presence of hydrogen in sintering atmosphere are favourable for decarburization effect in sintered steels.
7. The heterogeneous microstructure of investigated PM steels mainly consists of martensite or martensite+bainite (lower and upper).

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