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STRUCTURE AND MECHANICAL PROPERTIES OF Mn-Cr-Mo PM STEELS SINTERED IN DIFFERENT CONDITIONS

STRUKTURA I WŁASNOŚCI MECHANICZNE SPIEKANYCH STALI MANGANOWO-CHROMOWO-MOLIBDENOWYCH WYTWARZANYCH W RÓŻNYCH WARUNKACH

The effect of H_2/N_2 ratio in a sintering atmosphere on microstructure and mechanical properties of Fe-3%Mn-(Cr)-(Mo)-0.3%C PM steels has been shown. Pre-alloyed Astaloy CrL (1.5 wt.-%Cr and 0.25 wt.-% Mo) and Astaloy CrM (3 wt.-%Cr and 0.5 wt.-% Mo) powders were used to prepare the mixtures with low-carbon ferromanganese (77 wt.-% Mn and 1.3 wt.-% C) and graphite powders. Single-pressed compacts containing 3 wt.-% Mn and 0.3 wt.-% C with green density in the range of 6.8-7.1 $g\text{cm}^{-3}$ were produced. Sintering was carried out at 1120°C and 1250°C for 60 minutes under flowing different H_2/H_2 mixtures:

- A1 – 75 vol. % H_2 -25 vol. % N_2 ,
- A2 – 25 vol. % H_2 -75 vol. % N_2 ,
- A3 – 5 vol. % H_2 -95 vol. % N_2 ,
- A4 – 100 vol. % N_2 .

After isothermal sintering specimens were slow ($\sim 2^\circ\text{C}/\text{min}$) cooled to room temperature.

Tensile (UTS) and transverse rupture (TRS) strengths, elongation (A), $R_{0.2}$ yield offset, Young's modulus (E), impact toughness and apparent surface hardness were examined. Following mechanical tests, to investigate the microstructure of sintered Mn-Cr-Mo steels, light optical microscopy was employed.

It was shown that sintered properties are hardly influenced by the imposed sintering atmospheres. The best combination of plastic and tensile properties of examined steels was achieved after sintering in hydrogen-rich atmospheres.

Keywords: powder metallurgy (PM), PM steels, mechanical properties, sintering atmosphere, slow cooling, sintered microstructure, Young modulus

W pracy przedstawiony został wpływ składu chemicznego atmosfery spiekania, będącej mieszaniną wodoru i azotu, na strukturę i własności spiekanych stali o składzie chemicznym Fe-3%Mn-(Cr)-(Mo)-0.3%C. Do wytworzenia mieszanek proszków wykorzystano proszki stopowe Astaloy CrL (1,5% mas. Cr i 0,25% mas. Mo) i Astaloy CrM (3% mas. Cr i 0,5% mas. Mo) oraz niskowęglowy proszek żelazomanganu (77% mas. Mn i 1,3% mas. C). Założoną zawartość węgla uzyskano dodając do mieszanki proszków grafit. Wypraski o zawartości 3% mas. manganu i 0,3% mas. węgla, sprasowane jednostronnie w sztywnej matrycy, charakteryzowały się gęstością w zakresie od 6,8 g/cm^3 do 7,1 g/cm^3 . Spiekanie kształtek prowadzono w temperaturze 1120°C i 1250°C przez okres 60 minut, w atmosferze będącej mieszaniną wodoru i azotu o następujących proporcjach gazów składowych:

- A1 – 75% obj. H_2 -25% obj. N_2 ,
- A2 – 25% obj. H_2 -75% obj. N_2 ,
- A3 – 5% obj. H_2 -95% obj. N_2 ,
- A4 – 100% obj. N_2 .

Po spiekanu stalowe próbki chłodzone były do temperatury otoczenia wraz z piecem ze średnią szybkością $\sim 2^\circ\text{C}/\text{min}$.

W celu określenia wpływu warunków wytwarzania na strukturę i własności mechaniczne konstrukcyjnych, spiekanych stali manganowo-chromowo-molibdenowych przeprowadzono badania wytrzymałości na rozciąganie i wytrzymałości na zginanie jak również wyznaczono wydłużenie całkowite spieków oraz umowną granicę plastyczności $R_{0.2}$. Ponadto wykonano pomiary modułu Younga metodą ultradźwiękową, a także badania udarności oraz twardości spiekanych stali Mn-Cr-Mo. Identyfikację składników strukturalnych przeprowadzono w oparciu o badania metalograficzne spieków przy zastosowaniu mikroskopii świetlnej.

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Jak wykazały przeprowadzone badania, struktura i własności spiekanych stali manganowo-chromowo-molibdenowych silnie zależą od składu chemicznego atmosfery spiekania. Uzyskane wyniki pozwalają stwierdzić, że najlepszymi własnościami charakteryzowały się stale wytworzone w atmosferach bogatych w wodór.

Abbreviations

A	–	austenite;
At	–	elongation at fracture, %;
B	–	bainite;
C_{l2}	–	velocity of the longitudinal ultrasound waves;
E	–	Young's modulus, GPa;
E_1	–	elastic modulus along direction of pressing;
E_2	–	elastic modulus transversely to the direction of pressing along the sample width;
E_3	–	elastic modulus transversely to the direction of pressing, along the sample length;
d_0	–	green density, gcm^{-3} ;
d_1	–	as-sintered density, gcm^{-3} ;
HV ₃₀ surf.	–	apparent surface hardness;
M	–	martensite;
N/D	–	not defined;
ST	–	sintering temperature, °C;
$R_{0.2}$	–	0.2% offset yield strength, MPa;
TRS	–	transverse rupture strength, MPa;
UTS	–	ultimate tensile strength, MPa;

1. Introduction

The potential of Mn and Cr as alloying elements, reflected in their widespread use in wrought medium-to-high strength steels, is to be realised in powder metallurgy. An interesting development in the processing of PM steels is the addition of Mn to Fe-Cr-0.5%C steels sintered at $>1140^\circ\text{C}$ [1]. The mechanical properties of these steels were largely determined by the microstructure, so it is important to know how alloying elements affect hardenability and microstructural features. It is quite certain that the strengthening effect of Mn on Cr-containing steels is due to a combination of Mn and Cr present in the sintering atmosphere [2]. For the Astaloy CrL and CrM powders, containing 1.5 and 3% Cr, endogas is too wet for their processing. These steels have to be specially treated during sintering because of high affinity both of Mn and Cr for oxygen. Traditionally sintering atmosphere for low-alloy steel components is dissociated ammonia being substituted by synthetic mixtures of N_2/H_2 with a current industrial limit at 10% H_2 . To process Astaloy CrM-based steels industrially, however, methane is injected into nitrogen-hydrogen furnace atmosphere.

Cias et al [3] demonstrated that the problems of successful PM exploitation of steels containing ~3%Mn are associated with elimination of oxide networks, hitherto present in such alloys and, to prevent their formation, i.e. for MnO reduction, dew points of -55°C at 1120°C

and -40°C at 1200°C were necessary. It has also been shown [4, 5], that for successful processing of Mn containing steels it is essential that the dew point of the N_2/H_2 furnace atmosphere must be strictly controlled and be as low as possible. Only controlling the “local microclimate” ensured by use of semi-closed container or getter minimised interactions with the flowing atmosphere. Carbon monoxide (CO) generated within a PM component should prove to be a more efficient reducing agent than pure dry hydrogen at $>900^\circ\text{C}$. Also high temperature carbothermic sintering at $1250\text{--}1280^\circ\text{C}$ has been found important to produce the best combination of strength and ductility [6].

The purpose of current research was to control the effect of sintering atmosphere on the structure and mechanical properties of PM Mn-Cr-Mo structural steels.

2. Experimental methods, materials and procedures

Two groups of PM steels, based on pre-alloyed Höganäs Astaloy CrL (1.5 wt.-%Cr and 0.2 wt.-% Mo) and Astaloy CrM (3 wt.-%Cr and 0.5 wt.-% Mo) powders were manufactured and examined. The manganese powder was added as a low-carbon (1.3% C) ferromanganese (77% Mn), having particle size $12\ \mu\text{m}$ as measured by sedimentation method. Ultra fine graphite was also used (Figure 1).

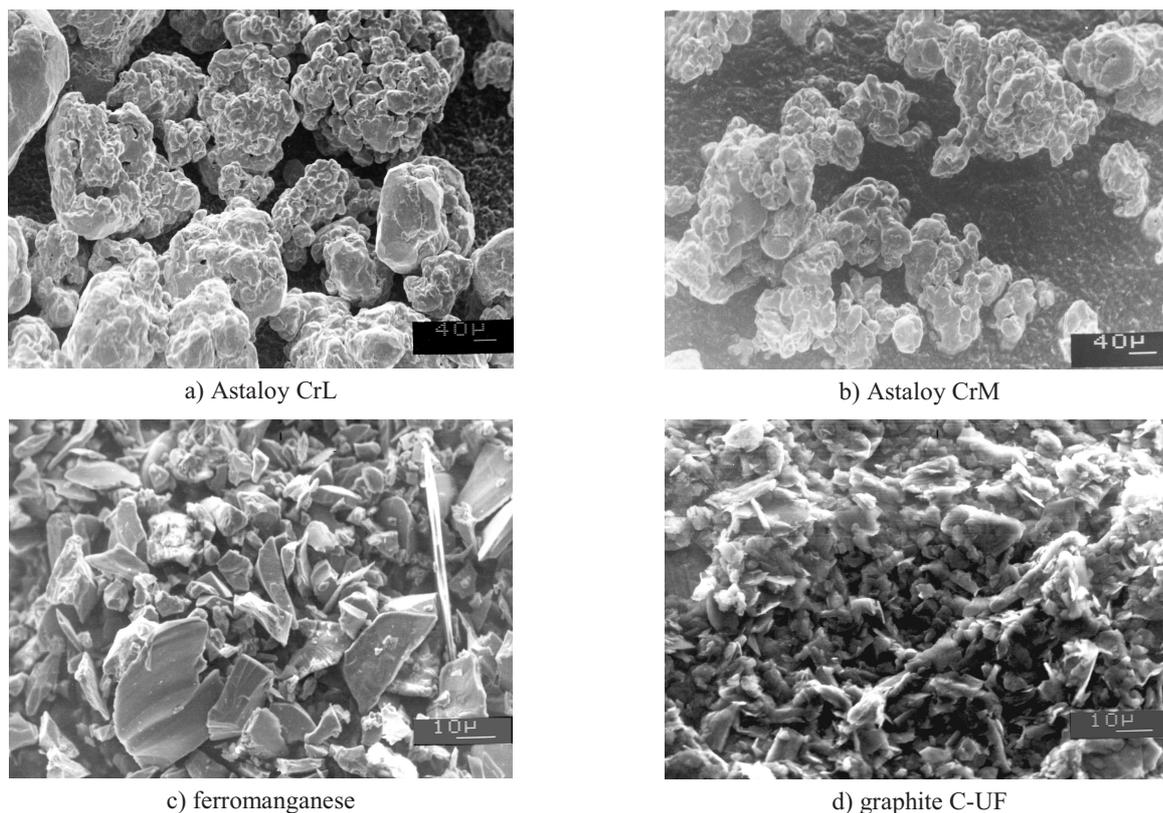


Fig. 1. SEM photography of the initial powders

Mixtures of powders containing 3% Mn, 0.3% C, 1.5 or 3% Cr and 0.2 or 0.5% Mo were prepared by blending ferromanganese, graphite and pre-alloyed powders in Turbula mixer for 30 minutes, to produce mixtures of the required uniform particles distribution. The blended powders were compacted in steel dies with zinc stearate lubricated walls. Uniaxial compacting with a stationary lower punch was used. Two types of compacts were prepared: rectangular transverse rupture test bars under pressing pressure of 820 MPa and tensile strength test bars under pressing pressure of 660 MPa. Test bars were prepared according to PN-ISO 5754 and ISO 2740 standard. The cold pressing pressure was adjusted individually to produce green compacts of nominally the same density. The green densities d_1 , are summarised in Table 1.

Isothermal sintering was carried out in 20 ppm moisture atmospheres with different H_2/N_2 ratio in Carbolite horizontal tube laboratory furnace type STF 15/75/610 at 1120 and 1250°C for 60 minutes employing slow ($\sim 2^\circ Cmin^{-1}$) cooling. The following sintering atmospheres were employed:

- A1 – 75% H_2 -25% N_2 ,
- A2 – 25% H_2 -75% N_2 ,
- A3 – 5% H_2 -95% N_2 ,
- A4 – 100% N_2 (by volume).

Average heating rate was 75°C/min and 70°C/min for sintering temperature 1120°C and 1250°C, respectively.

To improve the local dew point (self-gettering effect) and to minimise the loss of manganese due to volatilisation, the specimens were sintered in a semi-closed stainless steel container.

3. Testing of sintered specimens

The tensile properties were ascertained for as-sintered steels on “dogbone” tensile specimens according to the PN-EN 10002-1 standard. The tensile test was carried out with MTS 810 testing machine at a crosshead speed of 0.5 mm/min. The resulting stress-strain curves were analysed to identify the 0.2% offset yield strength ($R_{0.2}$), tensile strength (UTS) and tensile elongation at fracture (A). The Young’s modulus (E) was evaluated by the supersonic method along the investigated sample [7].

Transverse rupture strength (TRS) was determined by three-point bend test according to the PN-EN ISO 3325. The fixture had two support cylinders mounted parallel with a 28.6 mm distance between the centres. The testing equipment provided a static condition of loading. This procedure is only truly applicable to brittle

TABLE 1

Green and as-sintered density of Fe-Mn-Cr-Mo-C PM steels – mean values and standard deviation

PN-EN ISO 2740 – CrL+3%Mn+0.3%C					PN-EN PN-ISO 25754 – CrL+3%Mn+0.3%C				
d ₁ , gcm ⁻³	ST, °C	d ₂ , gcm ⁻³	ST, °C	d ₂ , gcm ⁻³	d ₁ , gcm ⁻³	ST, °C	d ₂ , gcm ⁻³	ST, °C	d ₂ , gcm ⁻³
6.92 ±0.02	1120	6.88 ±0.03	1250	6.91 ±0.03	7.12 ±0.05	1120	7.00 ±0.07	1250	6.96 ±0.07
ISO 2740 – CrM+3%Mn+0.3%C					PN-ISO 5754 – CrM+3%Mn+0.3%C				
d ₁ , gcm ⁻³	ST, °C	d ₂ , gcm ⁻³	ST, °C	d ₂ , gcm ⁻³	d ₁ , gcm ⁻³	ST, °C	d ₂ , gcm ⁻³	ST, °C	d ₂ , gcm ⁻³
6.86 ±0.03	1120	6.81 ±0.02	1250	6.81 ±0.05	6.95 ±0.05	1120	6.89 ±0.03	1250	6.96 ±0.07

TABLE 2

Mechanical properties of Fe-Mn-Cr-Mo-C PM steels – mean values and standard deviation

Sintering temperature and atmosphere		UTS, MPa	R _{0.2} offset, MPa	At, %	TRS, MPa	Impact toughness, J/cm ²	HV ₃₀ surf.
<i>CrL + 3% Mn + 0.3% C</i>							
1120	A1	512±60	428±90	1.3±0.4	907±91	3.2±1.5	190±29
	A2	438±47	361±35	0.9±0.4	1042±122	2.1±0.7	207±34
	A3	400±70	429±3	0.8±0.3	691±420	2.2±1.0	247±64
	A4	374±22	285±73	0.7±0.2	922±154	2.0±0.8	229±48
<i>CrM + 3% Mn + 0.3% C</i>							
1120	A1	584±59	498±32	1.1±0.4	987±63	3.1±1.0	255±46
	A2	532±32	451±67	1.1±0.4	1036±81	3.0±1.0	302±49
	A3	515±26	386±6	1.0±0.4	915±68	2.7±1.0	341±29
	A4	495±34	479±34	1.0±0.4	965±55	3.0±0.9	353±40
<i>CrL + 3% Mn + 0.3% C</i>							
1250	A1	602±50	398±61	1.6±0.4	1027±140	4.4±1.9	260±46
	A2	510±39	466±31	1.1±0.3	1214±83	4.5±1.8	249±63
	A3	456±79	484±53	0.8±0.4	1148±146	3.0±1.1	235±30
	A4	466±58	429±32	1.2±0.3	970±68	3.0±1.3	225±34
<i>CrM + 3% Mn + 0.3% C</i>							
1250	A1	816±68	587±16	2.5±0.7	1454±73	5.5±2.1	326±51
	A2	672±66	569±34	1.5±0.7	1153±69	3.6±1.4	345±40
	A3	572±163	526±22	1.1±0.3	1041±119	3.7±1.2	351±43
	A4	670±82	N/D	2.0±0.3	1061±84	2.9±1.0	362±32

materials [8]. It was used in this study as a routine measure quickly to distinguish between the apparent bend strength of investigated alloys.

Impact test was carried out using 55×10×5 mm specimens and a 15 J Charpy bar impact tester according to PN-EN 10045-1 standard.

The apparent surface hardness (HV₃₀ surf.) was determined by means of Vickers hardness tester according to PN-EN ISO 3878.

The as-sintered density, d₂, and mechanical properties of investigated PM steels are presented in Table 1 and 2, respectively.

Metallography investigations were carried out on polished and 3% nital-etched samples [9], using Leica DM4000M microscope.

4. Results

The properties of PM Fe-Mn-Mo-Cr-C steels are summarised in Tables 1 and 2. Both green and as-sintered densities were calculated using geometrical method. The results were verified by using Archimedes' method (Table 1).

Table 2 shows that for higher Cr and Mo concentration, high temperature sintering in hydrogen atmosphere has to be employed to improve strength properties. Ultimate tensile strength (UTS), elongation (A) and transverse rupture strength (TRS) after sintering at 1250°C were higher than those sintered at lower temperature, which correspond well to the bainitic/martensitic struc-

ture of PM steels. What is more, there is evident percentage differences between sintering in H₂-rich and pure N₂ atmosphere. For lower Cr and Mo concentrations, better properties were recorded after sintering in hydrogen-rich than in nitrogen atmosphere, irrespective to the sintering temperature. The same tendency as in strength properties was observed for plasticity and hardness of investigated steels (Table 2). Relatively high elongation can be also pointed out – 3.0% and 5.5% after sintering at 1120°C and 1250°C, respectively. High elongation values correspond well with the impact toughness.

Young’s modulus (E), evaluated by the supersonic method along the sample, was in the range of 144-170 GPa. The velocities of the longitudinal ultrasound waves along pressing direction were measured in three points (in both ends and in the middle) for each sample [7]. Figures 2 and 3 present the dependence of the C_{l2} values, measured in the middle points only, on the protective atmosphere used, for two samples made from Astaloy CrL and Astaloy CrM powders, respectively. The results of Young modulus are presented in Figures 4-9.

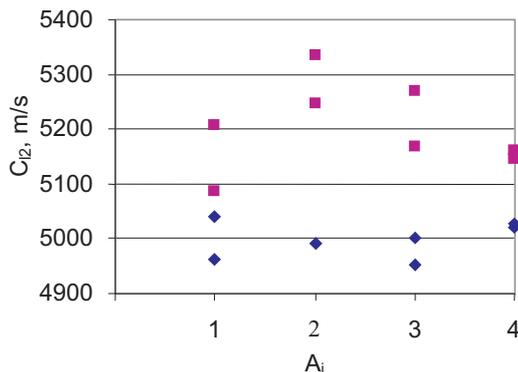


Fig. 2. Dependence of C_{l2} on the protective atmosphere for Astaloy CrL [7]

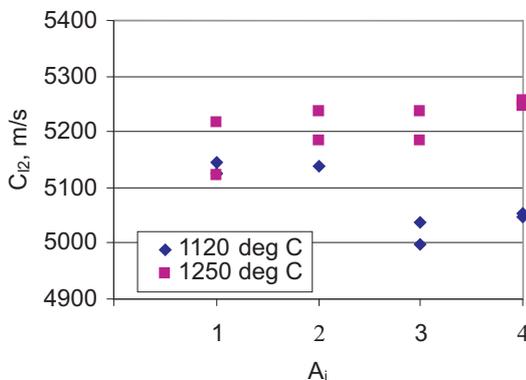


Fig. 3. Dependence of C_{l2} on the protective atmosphere for Astaloy CrM [7]

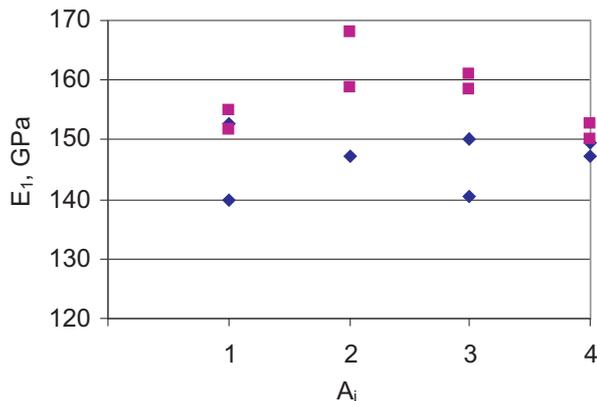


Fig. 4. Dependence of E_1 on the protective atmosphere for Astaloy CrL [7]

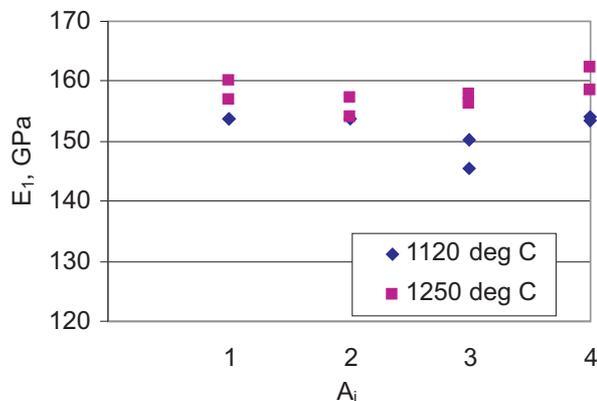


Fig. 5. Dependence of E_1 on the protective atmosphere for Astaloy CrM [7]

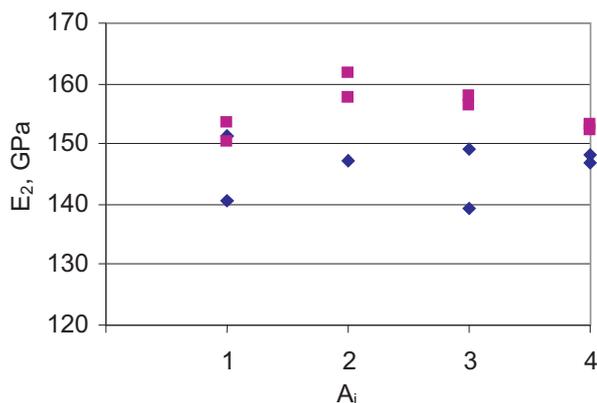


Fig. 6. Dependence of E_2 on the protective atmosphere for Astaloy CrL [7]

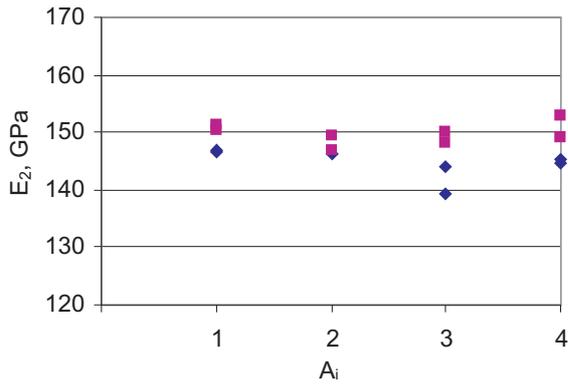


Fig. 7. Dependence of E_2 on the protective atmosphere for Astaloy CrM [7]

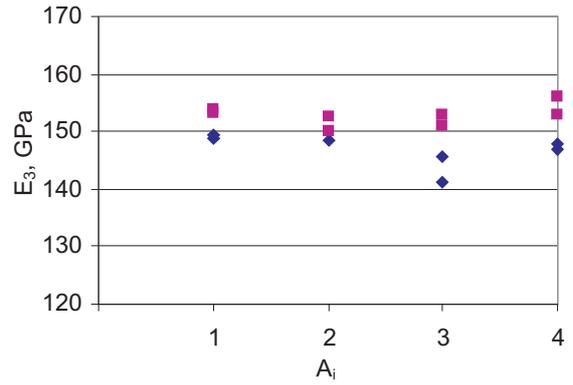


Fig. 9. Dependence of E_3 on the protective atmosphere for Astaloy CrM [7]

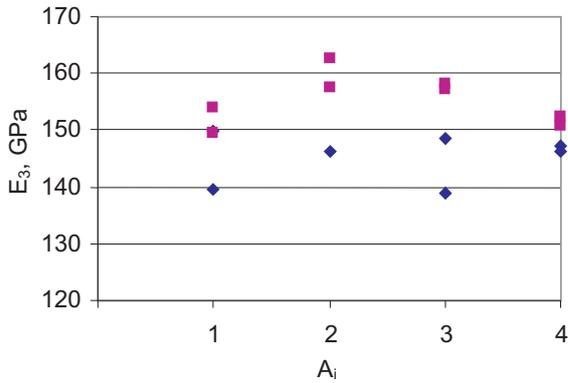


Fig. 8. Dependence of E_3 on the protective atmosphere for Astaloy CrL [7]

Figures 10-12 present the dependencies between the elastic moduli of investigated PM steels, obtained from the two pre-alloyed Astaloy powders in different experimental conditions. The results were statistically processed and approximated using linear relations. The obtained regression equations of high correlation ($R = 0.874 - 0.991$) are given in Table 3 [7].

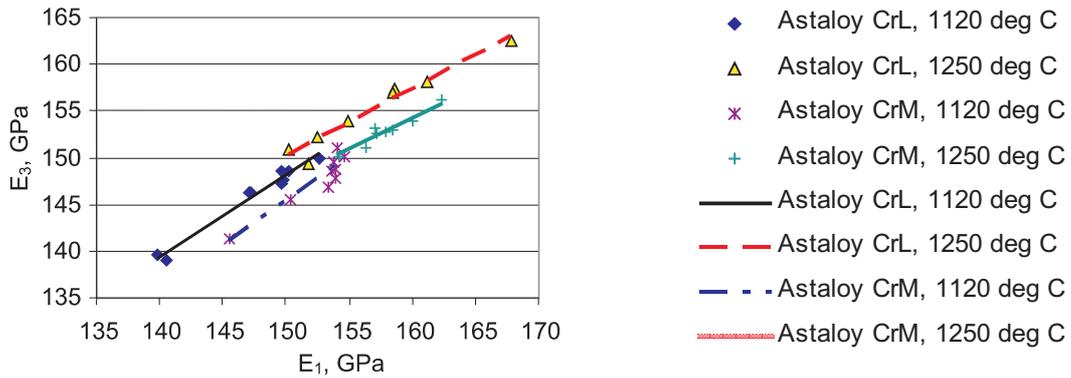


Fig. 10. Linear correlation between E_3 and E_1 for all experimental procedures applied [7]

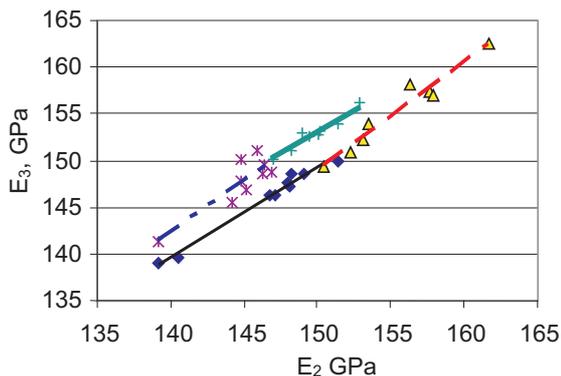


Fig. 11. Linear correlation between E_3 and E_2 for all experimental procedures applied [7]

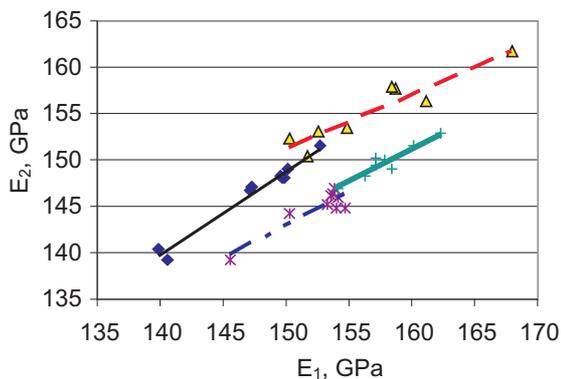


Fig. 12. Linear correlation between E_2 and E_1 for all experimental procedures applied [7]

TABLE 3

Regression equations for all experimental procedures applied [7]

Powder, grade	T, °C	Regression equations	R
Astaloy CrL	1120	$E_3 = 0.8723 E_1 + 17.318$	0.987
		$E_3 = 0.9598 E_2 + 5.2895$	0.991
		$E_2 = 0.9004 E_1 + 13.782$	0.987
	1250	$E_3 = 0.7276 E_1 + 40.983$	0.975
		$E_3 = 1.1644 E_2 - 25.715$	0.978
		$E_2 = 0.5878 E_1 + 63.099$	0.938
Astaloy CrM	1120	$E_3 = 0.9359 E_1 + 4.9509$	0.938
		$E_3 = 1.1073 E_2 - 12.636$	0.874
		$E_2 = 0.7190 E_1 + 35.144$	0.913
	1250	$E_3 = 0.6969 E_1 + 42.771$	0.959
		$E_3 = 0.9477 E_2 + 10.898$	0.971
		$E_2 = 0.6982 E_1 + 39.504$	0.938

The microstructural constituents of PM Fe-3% Mn-1.5%Cr-0.2%Mo-0.3%C and Fe-3%Mn-3%Cr-0.5% Mo-0.3%C steels are summarised in Table 4. For lower contents of Cr and Mo the structure of the material sintered at 1120°C consists of bainitic/martensitic regions. The effect of increasing Cr and Mo level was that a mainly martensitic/austenitic structure was observed.

After sintering at 1250°C, the structure consists mainly of martensite and bainite regions with small amount of austenite. After sintering at 1120°C the carbon levels in Astaloy CrL and Astaloy CrM-based materials was decreased to 0.27%, whereas in the materials sintered at 1250°C the carbon contents were reduced to 0.19 and 0.21%, respectively. Figures 13-16 present characteristic microstructure of investigated steels.

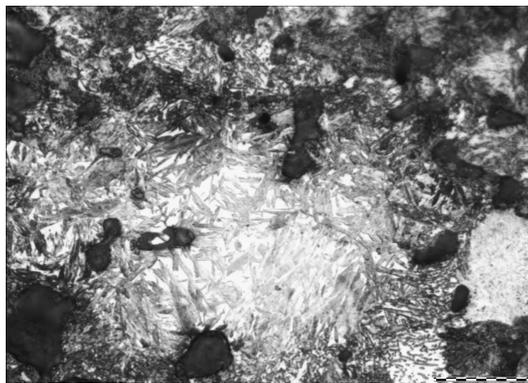


Fig. 13. Martensitic/austenitic structure of Astaloy CrL-based steels; 1120°C/N₂/1000x

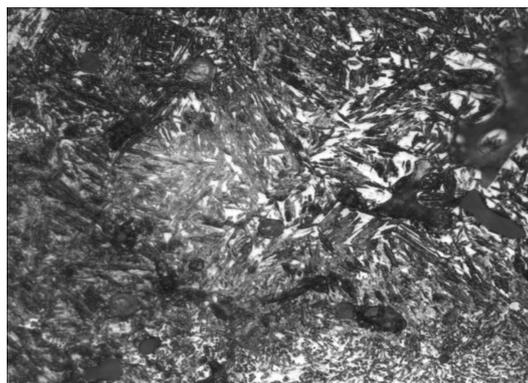


Fig. 14. Martensitic/bainitic/austenitic structure of Astaloy CrM-based steels; 1120°C/N₂/1000x

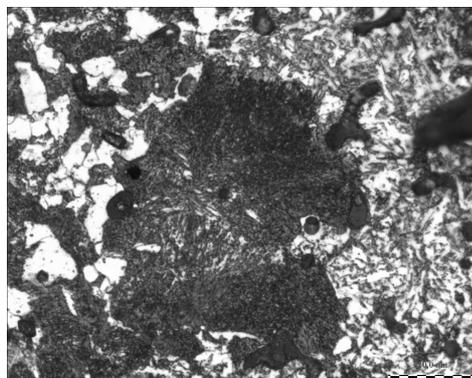


Fig. 15. Bainitic/martensitic structure of Astaloy CrL-based steels; 1250°C/N₂/1000x

Structural constituents identified in investigated steels

ST	ATM	<i>CrL+3%Mn+0.3%C</i>	<i>CrM+3%Mn+0.3%C</i>
T1	A1	B +M +A	B or M +A
	A2	B or B + small M +A	M + A, B islands very seldom
	A3	B +M +A, F islands	M + A, small amount of B
	A4	M + A	M +A + B
T2	A1	B / M+B+A, isolated B islands	M + B+A / B
	A2	B, small M+A	M+A, B islands
	A3	B small M+A or F islands	M+A, small B
	A4	B / B+M, small A	M+A

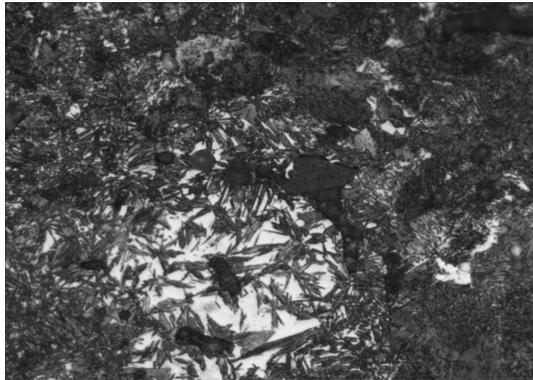


Fig. 16. Martensitic/austenitic/bainitic structure of Astalloy CrM-based steels; 1250°C/N₂/1000x

5. Summary and conclusions

The work carried out has described the possibility of producing Fe-Mn-Cr-Mo-C PM steels in other than a hydrogen atmosphere. The author was interested in the nitrogen-sintering route for several reasons. Not the least of which is that the EU has a wish to reduce H₂ levels in furnace gases to a level less than that which is explosive (<4%) [10–12]. It was therefore felt necessary to study the influence of a type of sintering atmosphere on the utilisation of Mn and Cr additions to obtain better economical results and to take suitable safety precautions. The work was carried out using N₂/H₂ atmosphere with a dew point of <−60°C (~ 20 ppm moisture), so that it is rather difficult to make a direct comparison with results obtained in industry, and with other published work, where different N₂/H₂ atmospheres had been used. The dew point refers to the dryness of a H₂ only atmosphere; if diluted (given water vapour content), the reducing potential is reduced, because amount of H₂ is reduced. In flowing N₂ 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/CO₂ ratio, ensures optimum conditions for thermo-carbic oxide reduction and efficient sintering [2]. Specimens sintered at 1250°C possessed higher mechanical properties, irrespective of the H₂-N₂ ratio in the furnace atmosphere. For the investigated steels the tensile (UTS) and transverse rupture strength (TRS) were approximately directly proportional to the H₂ content in the sintering atmosphere. Chromium enhances, however, as compared with previous results [4], the detrimental effects of N₂ on the strength of the manganese steels. This effect can be confirmed by chemical analysis in investigated steels. Nitrogen in investigated steels improves hardness and has little effect upon elongation and impact toughness [4]. The effect of N₂ on brittle fracture of these steels should be investigated further, especially in regard to the “clustering” N₂ atoms about substitutional alloy atoms Cr and Mn.

Regarding microstructures of the investigated steels, N₂ with a dew point of −60°C did not prove as successful a furnace atmosphere as equally dry H₂. It is therefore concluded that, provided sintering of Fe-3%Mn-1.5%Cr-0.2%Mo-0.3%C and Fe-3%Mn-3%Cr-0.5%Mo-0.3%C steels is carried out in a semi-closed container (with availability of carbon and manganese therein, in our case within the green compact), a furnace atmosphere of dry N₂ is not as effective in preventing formation of deleterious oxide networks as of dry H₂, or of H₂-N₂ mixtures.

The *C*₁ values reveal longitudinal inhomogeneity of the sintered steels, which should be taken into consideration in the analysis of the Charpy test data.

The reduction potential increases with the increase of hydrogen content in the protective atmosphere, i.e.: oxide concentration in the material will be lower and thus higher strength of the PM materials could be expected, while nitrogen has little influence on the oxide concentration. The lowest scattering of the *C*₁₂ and the elastic moduli *E* values in the sample sets was observed for protective atmospheres, as was shown in Figures 2-3.

A set contains all the samples prepared from the same powder, sintered under the same conditions (temperature, atmosphere).

The E values of PM structural steels, based on Astaloy CrL powder, sintered at $T = 1250^{\circ}\text{C}$ in a mixture of 5% H_2 -95% N_2 , show slight scattering, while a maximum is observed for the samples sintered in atmosphere containing 25% H_2 and 75% N_2 . The lowest E values were obtained for steels with higher chromium and molybdenum content after sintering at 1250°C in pure nitrogen.

Although the composition of material has little effect on E , it could be seen in Figures 4-12, that the elastic modulus of low-chromium, low-molybdenum PM steels, sintered at 1250°C is slightly higher than for the samples made from Astaloy CrM powder, sintered at the same temperature.

The following conclusions can be drawn from the results of the performed study:

1. Mechanical properties of investigated steels are rather high. The highest properties were achieved for steels sintered both in H_2 -rich atmosphere and in H_2/N_2 mixtures.
2. The structure of PM Mn-Cr-Mo steels consists mainly of martensite, bainite and austenite. The percentage of microstructural constituents depends on the chemical composition of steel, type of sintering atmosphere and sintering temperature.
3. The high frequency ultrasound echo-pulse method can be used to determine the homogeneity of PM samples that are to be subjected to mechanical tests.
4. A slightly pronounced anisotropy (up to 1-2%, related to E_1), typical for PM parts, was observed after taking into account the Rayleigh corrections and calculating the elastic moduli E_1 , E_2 and E_3 .
5. The type of the protective atmosphere influences mainly the stability of the elastic moduli.

6. The elastic moduli values increase with the increase of the sintering temperature from 1120°C to 1250°C .

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