Tensile properties of Fe–3Mn–0·6/0·7C steels sintered in semiclosed containers in dry hydrogen, nitrogen and mixtures thereof

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Tensile properties of powder metallurgy 3% manganese–0·8% carbon (content of green compact) steels were determined following laboratory sintering in (nearly) full, semiclosed containers with no getter powders in dry, 0–100% hydrogen–nitrogen atmospheres. Manganese was mixed with the NC 100·24 sponge iron powder as low carbon ferromanganese and carbon as a graphite addition. Dogbone compacts were pressed at 660 MPa, the sintering temperatures were 1120 and 1250°C and cooling rates $\sim$65 K min^{-1}. In specimens sintered in nitrogen containing atmospheres at 1120°C, final carbon content was $\sim$0·7% and for those processed at 1250°C $\sim$0·6%. Sintering in dry hydrogen resulted in lower carbon and oxygen contents. Independent of the H_2/N_2 ratio in the furnace atmosphere, however, all the specimens were ductile and exhibited similar strengths. Yield strengths $R_{0.2}$ were in the range: 426–464 MPa, tensile strengths R_m were 724–780 MPa and strains to failure were 1·6–2·0% after sintering at 1250°C. The 1120°C sintering temperature resulted in 10–15% lower strength values. The microstructures, significantly devoid of oxide networks, comprised mainly mixtures of bainite and fine (divorced) pearlite, with very little martensite and retained austenite.

Reproducibly successful sintering of manganese containing compacts requires that reduction conditions exist at the sintering temperature. Ellingham–Richardson diagrams dictate that the dewpoints of hydrogen required are -55 and $-40°C$ at 1120 and 1250°C, respectively. A semiclosed container, however, ensures a different microclimate. It is suggested that then the initial relevant reactions there are: $Mn[vapour] + H_2O = MnO + H_2$, $3Fe_2O_3 + H_2 = 2Fe_3O_4 + H_2O$, $Fe_3O_4 + H_2 = 3FeO + H_2O$, $FeO + H_2 = Fe + H_2O$ and $C + O_2 = CO_2$, which provide hydrogen and water vapour, also within the pores. The manganese vapour further acts as a 'shield' by generating further hydrogen from the water vapour. The following reactions involving carbon monoxide are postulated above 927°C, when CO is a more effective reducing agent than hydrogen: $C + H_2O = H_2 + CO$, $3Fe_2O_3 + CO = 2Fe_3O_4 + CO_2$, $Fe_3O_4 + CO = 3FeO + CO_2$, $FeO + CO = Fe + CO_2$ and $C + CO_2 = 2CO$. Accordingly, irrespective of whether it is hydrogen or nitrogen in the semiclosed container, if there is a supply of carbon, reducing conditions prevail at the sintering temperature, embrittling oxide networks are not formed and ductile manganese steels are processed. PM/1033

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INTRODUCTION

Sintering of manganese steels has been extensively researched by Salak, who also first recognised that manganese is volatile and accordingly, in a compact with open porosity, Mn vapour transport is the dominant diffusion mechanism in the sintering process of these steels. His most recent analysis is presented in Ref. 1. He and other early researchers,[2–6] however, failed to obtain adequate reproducible mechanical properties and commercial exploitation appears restricted to <1·5%Mn alloys.[7] It was only in 1998 that Cias, Mitchell and Wronski[8–11] demonstrated that the problems of successful PM exploitation of steels richer in Mn are associated with the formation of oxide networks, hitherto present in such experimental alloys. In a European Union COPERNICUS project,[8] probably for the first time, 3 and 4%Mn steels free of oxide networks have been processed and prototype gears were successfully sintered in semiclosed containers.

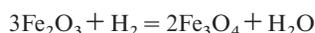When laboratory experiments were carried out in a flowing hydrogen rich gas, it was found that dewpoints dictated by an Ellingham–Richardson diagram for Mn–O reduction were necessary, namely $-55°C$ at 1120 and $-40°C$ at 1200°C. It should be recalled that dewpoint refers to the temperature at which moist air becomes saturated and therefore indirectly the water vapour content in the furnace atmosphere. What is important regarding oxidation/reduction during the sintering cycle is the reducing potential; for pure H_2 at any temperature it is proportional to the ratio of partial pressures of H_2O to H_2: 5×10^{-5} at 1120°C and 10^{-4} at 1250°C. For an atmosphere of pure hydrogen there is a simple relationship between this ratio and water vapour content and therefore the dewpoint. If an hydrogen atmosphere is diluted with other gases e.g. by addition of nitrogen, then, for a given water vapour content, i.e. displayed dewpoint, the reducing potential is lowered because the amount of H_2 is reduced. Thus dewpoint meter readings of $-40°C$ for 3:1 and 1:3 hydrogen/nitrogen mixtures are degraded effectively by $\sim$4 and $\sim$13°C, respectively. For hydrogen free atmosphere it becomes $\sim$40°C and flowing dry nitrogen atmosphere does not prevent oxidation during sintering. As oxides are reduced and water vapour forms, it is further important that both a low inlet gas
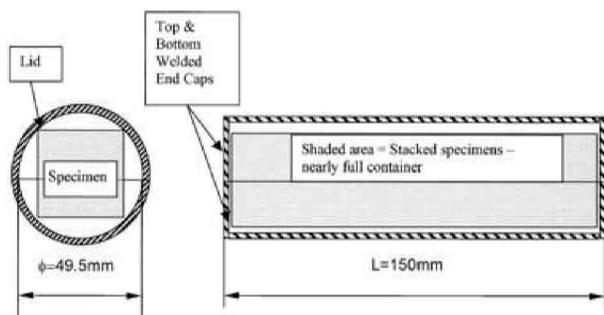
1　Schematic of split tube type semiclosed stainless steel container used for sintering experiments

dewpoint and an adequate gas flow are maintained in order to minimise potentially oxidising water vapour in contact with the specimens during sintering ('specimen microclimate').

Only variation in the local microclimate in a semiclosed container/getter can affect the, thermodynamically governed, reactions:

$$Mn + H_2O = MnO + H_2$$

$$3Fe_2O_3 + H_2 = 2Fe_3O_4 + H_2O$$

$$Fe_3O_4 + H_2 = 3FeO + H_2O$$

$$FeO + H_2 = Fe + H_2O$$

$$MnO + H_2 = Mn + H_2O$$

$$C + O_2 = CO_2$$

$$C + CO_2 = 2CO$$

Accordingly, additionally with regard to the safety and cost of lean hydrogen sintering atmospheres and legislation defining hydrogen always as a tax attracting fuel, it was decided to vary the hydrogen content from zero to 100% in dry hydrogen–nitrogen sintering atmospheres. In the absence of sufficient hydrogen, the selfgettering properties of manganese and carbon in the green compacts of manganese steels were thus being investigated.

EXPERIMENTAL PROCEDURES
Processing
Höganäs sponge, NC100·24, iron powder was used for this investigation. 0·8% of carbon was introduced as fine graphite and 3% of manganese as Elkem low carbon ferromanganese. This latter powder is a by product, 'fines', from electrode production, of wt-% composition 79·3Mn–1·2C–0·4Si–2·3O–0·1N–Fe(Bal.), which was milled before sieving to less than 20 μm with a median of 8 μm.

Mixing and die compaction, using only die lubrication, at 660 MPa of ISO 2740 dogbone specimens were followed by sintering in nearly full semiclosed stainless steel containers (*see* Fig. 1) pushed into the hot zone of a Kanthal APM horizontal tube furnace. At least 10 tensile specimen compacts were simultaneously sintered and the remaining space in the container was filled with ferrous ballast or other specimens of identical composition. The furnace atmospheres were 100, 75, 25 and 5% hydrogen, balance nitrogen and nitrogen only. The inlet dewpoint of the gases was maintained below $-60°C$. The gas flowrate was about $1 m^3 min^{-1}$. Sintering times were 1 h and sintering temperatures 1120 and 1250°C. There was no significant change in density from green values ($\sim 6·9 g cm^{-3}$). After sintering, the specimens were withdrawn from the hot zone and allowed to cool in the cool zone of the furnace. Approximate heating and cooling to 300°C rates were 75 and $\sim 65 K min^{-1}$ respectively.

Analytical, metallographic and microstructural examinations
Conventional metallographic techniques were supplemented by scanning electron microscopy, generally on a JEOL 6400 instrument fitted with a KEVEX Sigma 3 energy dispersive X-ray microanalysis system, which enabled fairly accurate determinations of microcomposition. Chemical analyses for oxygen, carbon and nitrogen were carried out on a Leco apparatus, TC–336 and CS–125.

Tensile testing
Standard ISO 2740 tensile specimens were tested on an MTS 810 servohydraulic machine at extension rates of $\sim 5 mm min^{-1}$.

RESULTS
Analytical data
Using Leco equipment, triplicate analyses were carried out for carbon, oxygen and nitrogen for each batch of specimens. The results are presented in Table 1. The data indicate that, if there is nitrogen in the furnace atmosphere, in the sintered specimens:
- there is a carbon loss from 0·8% added of approximately 0·1 and 0·2% after processing at 1120 and 1250°C, respectively
- oxygen contents are approximately 0·5% after sintering at 1120°C and lower, at 0·3–0·4%, for the 1250°C processing.

For specimens sintered in dry hydrogen, oxygen and carbon contents are lower; most significant being the $\sim 0·15\%$ oxygen figure for the 1250°C sintering temperature. Nitrogen contents increased with increasing nitrogen content of the atmosphere, from $\sim 0·003$ hydrogen only to 0·03% nitrogen only.

Table 1　Carbon, oxygen and nitrogen analyses for all batches of sintered specimens

Sintering conditions	Carbon, wt-%	Oxygen, wt-%	Nitrogen, wt-%
1120°C, H_2 only	0·627	0·316	0·0036
	0·634	0·303	0·0035
	0·641	0·311	0·0036
1120°C, 75%H_2–25%N_2	0·720	0·513	0·0135
	0·698	0·381	0·0126
	0·714	0·463	0·0129
1120°C, 25%H_2–75%N_2	0·729	0·466	0·0247
	0·730	0·491	0·0242
	0·730	0·535	0·0270
1120°C, 5%H_2–95%N_2	0·757	0·507	0·0296
	0·742	0·496	0·0291
	0·749	0·464	0·0304
1120°C, N_2 only	0·726	0·606	0·0327
	0·704	0·588	0·0331
	0·712	0·584	0·0339
1250°C, H_2 only	0·496	0·130	0·0032
	0·532	0·162	0·0031
	0·530	0·151	0·0033
1250°C, 75%H_2–25%N_2	0·575	0·412	0·0259
	0·585	0·307	0·0274
	0·580	0·256	0·0257
1250°C, 25%H_2–75%N_2	0·568	0·360	0·0278
	0·567	0·361	0·0239
	0·572	0·254	0·0252
1250°C, 5%H_2–95%N_2	0·620	0·271	0·0333
	0·618	0·281	0·0327
	0·621	0·295	0·0323
1250°C, N_2 only	0·624	0·318	0·0336
	0·615	0·384	0·0335
	0·617	0·327	0·0302
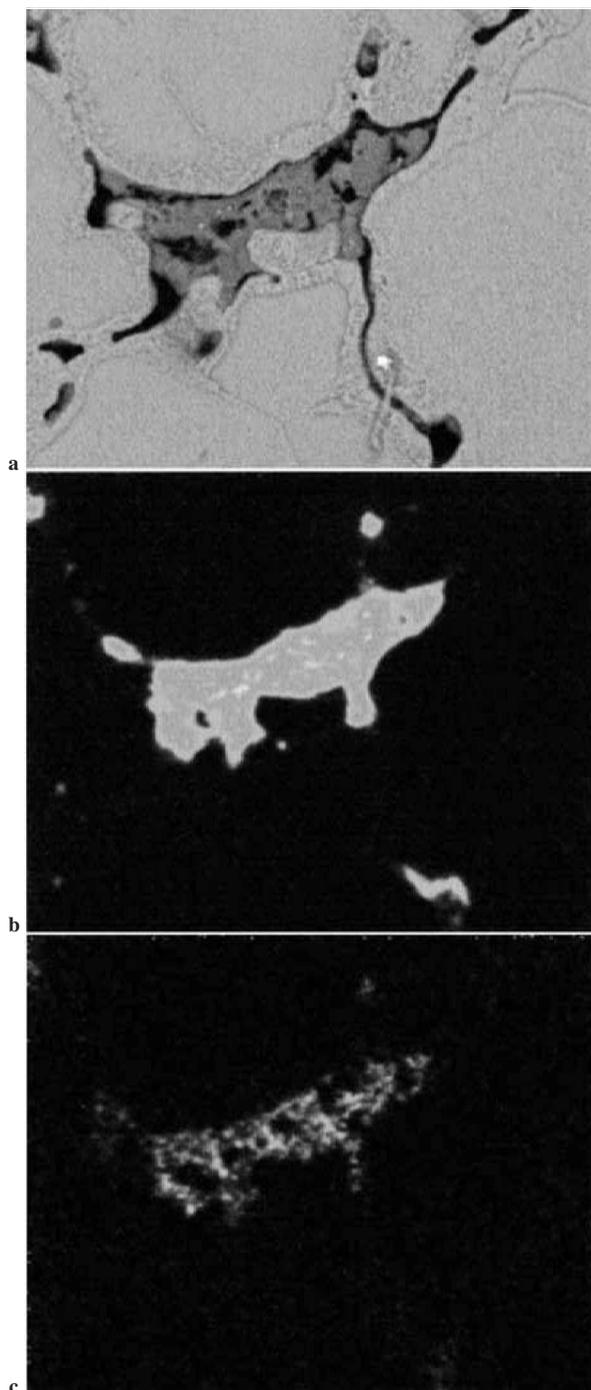
a SEM image; *b* Mn X-ray dot map; *c* O X-ray dot map

2 Typical oxide network in Fe–3%Mn steel sintered at 1120°C in poor dewpoint of −15°C

Metallographic and microstructural observations

Until recently, presence of manganese oxides was characteristic of PM manganese steels,[4–8] an example of which is presented in Fig. 2. The most relevant feature of the microstructures was the absence of oxide networks (e.g. Fig. 3), whatever the proportions of nitrogen and hydrogen in the sintering atmosphere. All microstructures comprised a mixture of bainite (predominantly) and fine (divorced) pearlite (troostite), with very little martensite and retained austenite. In the surface layers very little ferrite was observed. There was evidence of decarburisation in specimens sintered in hydrogen only. The pores were more rounded in specimens sintered at 1250°C. Areas of high manganese retained austenite were present around larger pores due to the

transport of manganese in the vapour phase. Mn diffusion distance was determined to be of the order of 15 μm at sintering temperatures <1150°C and times of ~1 h.[11]

Mechanical properties

The values of Young's modulus, 0·2% offset yield and tensile strengths and failure strain were determined for 20 specimens of each batch. The results were averaged and are presented in Table 2.

DISCUSSION
Mechanical properties

The data presented in Table 2 reproducibly and consistently indicate that:
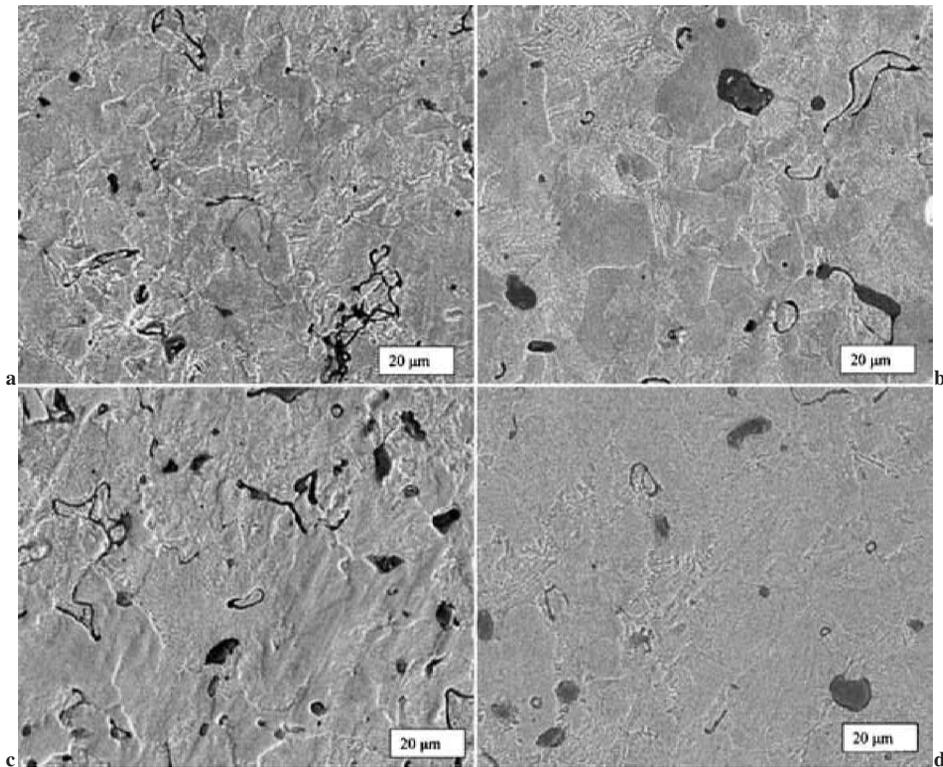
- there is no significant influence of the tensile properties on the composition of the sintering atmosphere for a given sintering temperature
- strengths are somewhat higher, 10–15%, after processing at 1250°C than at 1120°C
- the specimens exhibit between 1·6 and 2·0% tensile strain before failure.

Sintering atmospheres

The mechanical properties indicate that, provided sintering of a Mn–C steel is carried out in a semiclosed container (with availability of carbon and manganese therein), it matters little whether the furnace atmosphere is dry hydrogen or dry nitrogen. The interpretation of successful sintering in an atmosphere of dry hydrogen, in open or closed containers,[8–13] can rely on the reducing properties of hydrogen, governed by the Ellingham–Richardson diagram. Reduction with hydrogen creates water vapour, which leads to the observed decarburisation. Additionally, more carbon will be consumed when it reacts with any generated CO_2 to form CO (above 927°C) and with H_2 to form methane (above ~1200°C). Presence of hydrogen in the H_2–N_2 sintering atmospheres will allow similar reactions to take place. Even just a 5% addition of hydrogen lowers residual oxygen without creating a large carbon loss for both sintering temperatures. The success of nitrogen sintering atmosphere in preventing oxidation of manganese, in the microclimate of the semiclosed container, however, requires further consideration.

Table 2 Mean values of Young's modulus E, yield strength $R_{0·2}$, tensile strength R_m and strain to failure ε [total] for 10 batches of specimens sintered in dry hydrogen, nitrogen and combinations thereof
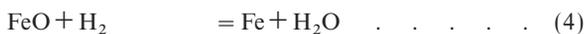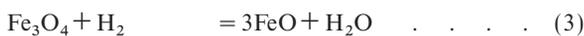
Sintering conditions	E, GPa	$R_{0·2}$, MPa	R_m, MPa	ε [total], %
1120°C, H_2 only	114	386	633	1·8
	±5	±23	±59	±0·4
1120°C, 75%H_2–25%N_2	115	400	639	1·8
	±6	±24	±39	±0·3
1120°C, 25%H_2–75%N_2	120	420	648	1·8
	±5	±13	±35	±0·3
1120°C, 5%H_2–95%N_2	122	421	654	1·8
	±4	±17	±32	±0·2
1120°C, N_2 only	117	410	652	1·9
	±6	±12	±30	±0·2
1250°C, H_2 only	117	426	724	1·6
	±6	±26	±39	±0·1
1250°C, 75%H_2–25%N_2	117	443	741	1·7
	±4	±16	±37	±0·2
1250°C, 25%H_2–755N_2	120	459	780	1·8
	±4	±12	±28	±0·2
1250°C, 5%H_2–95%N_2	118	451	728	1·6
	±5	±21	±74	±0·3
1250°C, N_2 only	122	464	776	2·0
	±5	±22	±33	±0·2

3   Scanning electron micrographs of specimens sintered at *a* 1120°C in 100% nitrogen atmosphere; *b* 1250°C in 100% nitrogen atmosphere (note the rounder pores compared to part *a*); *c* 1120°C in 100% hydrogen atmosphere to give lower final oxygen content than specimen in part *a*; *d* 1250°C in 100% hydrogen atmosphere (note rounder pores and lower oxygen content than specimen in part *c*)

It should be stressed that, however dry the gas, there is always some water vapour present to react with the metals, that Mn is volatile and that there is also a 'supply' of carbon in the green compact. Reference should also be made to the recent report of Dudrova *et al.* on sintering of Mn steels in 25%H$_2$–75%N$_2$ atmosphere carried out either in flowing gas or in semiclosed containers with a getter of alumina, graphite and ferromanganese.[13,14] Interestingly, they found losses of carbon and manganese during sintering in the flowing gas and gains in the contents of both elements when sintering took place in semiclosed containers. Reactions involving carbon, manganese and water vapour thus need to be considered, not forgetting that the major metallic element is iron.[15] The possibility of an oxide of manganese (as is true for ruthenium and titanium) acting as a catalyst for the carbon reactions should not be discounted. The thermodynamic aspects of reactions with carbon and carbon monoxide of iron, manganese and chromium are considered by Mitchell[16] in a companion paper to this one.

It is suggested that the initial relevant reactions are

$$Mn[vapour] + H_2O = MnO + H_2 \qquad \qquad (1)$$

$$3Fe_2O_3 + H_2 \qquad = 2Fe_3O_4 + H_2O \qquad (2)$$

$$Fe_3O_4 + H_2 \qquad = 3FeO + H_2O \qquad (3)$$

$$FeO + H_2 \qquad = Fe + H_2O \qquad (4)$$
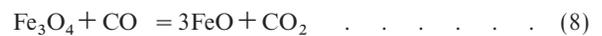
$$C + O_2 \qquad = CO_2 \qquad \qquad (5)$$
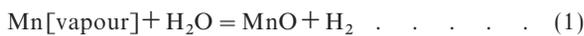
These reactions provide hydrogen and water both within the pores and in the semiclosed container. Manganese vapour further acts as a 'shield' by generating further hydrogen from the water vapour. The generated hydrogen continues to reduce the oxides of iron, in effect a 'hydrogen reduction loop' is created. Now consider CO generation

$> 927°C$

$$C + H_2O \qquad = H_2 + CO \qquad \qquad (6)$$

and therefore

$$3Fe_2O_3 + CO = 2Fe_3O_4 + CO_2 \qquad (7)$$

$$Fe_3O_4 + CO \quad = 3FeO + CO_2 \qquad (8)$$

$$FeO + CO \quad = Fe + CO_2 \qquad \qquad (9)$$

also the Boudouard reaction

$$C + CO_2 \qquad = 2CO \qquad \qquad (10)$$

This and reaction (6), lead to C loss.
The net reaction of (9), (10) and (6) gives

$$FeO + C \qquad = Fe + CO \qquad \qquad (11)$$

It should be noted that

$$\frac{partial\ pressure\ H_2}{partial\ pressure\ H_2O} = K \frac{partial\ pressure\ CO}{partial\ pressure\ CO_2}$$

To also be considered are these possible reactions

$$3Fe + CO + H_2 \qquad = Fe_3C + H_2O \qquad (12)$$

$$3Fe + 2CO \qquad = Fe_3C + CO_2 \qquad (13)$$

$$3Fe + C \qquad = Fe_3C \qquad \qquad (14)$$

$$MnO + CO \qquad = Mn + CO_2 \qquad (15)$$

$$2C + O_2 \qquad = 2CO \qquad \qquad (16)$$
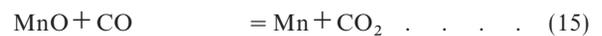
and the Sabatier reaction

$$CO_2 + 4H_2 \qquad = CH_4 + 2H_2O \qquad (17)$$

Another reaction leading to C loss is the methane reaction $> 1200°C$
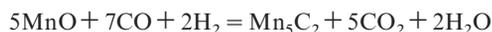
$$2H_2 + C \qquad = CH_4 \qquad \qquad (18)$$

For temperatures over 1200°C Salak *et al.*,[1] drew attention to these overall reduction reactions[17] by the carbide route

$$5MnO + 7C \qquad = Mn_5C_2 + 5CO \quad . \quad . \quad . \quad (19)$$

$$2MnO + Mn_5C_2 \qquad = 7Mn[vapour] + 2CO \qquad (20)$$

But,

$$MnO + C \qquad = Mn + CO$$

only react directly above ~ 1400°C (consider blast furnace reactions), so a possibility now suggested is

$$5MnO + 7CO + 2H_2 = Mn_5C_2 + 5CO_2 + 2H_2O$$

and/or a complicated reaction using products from reactions (6) and (10), leading to further reduction

$$2MnO + Mn_5C_2 \qquad = 7Mn[vapour] + 2CO$$

Accordingly the higher carbon loss after sintering at 1250°C is attributed to the Boudouard, the methane forming and the reduction of MnO by carbide reactions.

Formation of MnO on the inside of pores on cooling is benign and some oxygen possibly emanates from inclusions such as silicates, which were not successfully sieved out from the ferromanganese.[13] Lower oxygen contents appear related to the higher sintering temperature. At ~ 0.4% content oxygen did not prevent ductility reproducibly exceeding 1·5% and no evidence of oxide networks was found by scanning electron microscopy.

It should be added that

- hydrogen is wetter than nitrogen
- in pure nitrogen, the redox line is crossed at higher temperatures, hence more MnO is then formed (probably benign inside pores and on the surface)
- above 1000°C, CO_2 concentration is negligible and below 400°C, the reverse is true
- CO becomes a more efficient reducer than H_2 above 900°C
- MnO is probably the only relevant oxide as higher oxides require long times and high temperatures to be formed from MnO in air.

The determination of the atmospheres, with varying composition and temperature, awaits mass spectrometric experiments, which are currently not possible at either of the authors' laboratories. In the absence of knowledge of, for each set of sintering conditions, the distribution of the total oxygen and its forms, the amount of benign manganese oxide in the specimens, how much manganese was lost by evaporation, how much carbon was consumed in reactions with oxides, how much to form CO (Boudouard reaction) and how much to form methane at the higher sintering temperature, it is not realistic to offer an interpretation of the absolute values of carbon and oxygen contents. Not only is it not possible, but probably undesirable, as ultimately the 'acceptable' range of processing parameters and resultant carbon and oxygen contents is of industrial importance. It should be added that the manganese and carbon 'supply' could then be provided in the form of a getter powder in the semiclosed container, and results of Dudrova *et al.*[13] indicate that then there need not be any loss of carbon or manganese.

Nevertheless it is relevant to this communication to indicate the concentration ranges of carbon and oxygen in specimens nominally identically processed. Table 3 presents the data for specimen batches processed in the same furnace in hydrogen in the temperature range 1120–1200°C with an inlet dewpoint of -60°C. It is evident that, whereas for a given batch, there is very good reproducibility of the analyses data, the difference in analyses between batches nominally identically processed is larger. One of the parameters, kept constant for this set of experiments, influencing slightly the precise compositions of oxygen and carbon has been shown to be the cooling rate from the sintering temperature probably concentrations within ± 0.05% are

meaningful. Regarding the nitrogen contents, increasing to 0·03 from 0·003%, as the atmosphere became only nitrogen, these are considered of secondary importance, other than possible surface nitriding in nitrogen rich atmospheres on cooling. Nitrogen content, even at 0·03%, is insignificant compared with carbon at over 0·5%. Whether it replaces or is added to carbon interstitially or to form carbonitride(s), it should not matter.

To investigate the validity of the 'carbon' hypothesis for pure nitrogen sintering of manganese steels in semiclosed containers, a carbon free Fe–3Mn (added as electrolytic) alloy was sintered at 1120°C in a Leco crucible and analysed for oxygen. The content increased from 0·30 to 0·456% (mean of five determinations), in contrast to a similar, independent, experiment with Fe–3Mn–0·8C, in which it decreased to 0·279%, with a concurrent loss of carbon to 0·519%. For successful sintering of manganese steels in dry nitrogen, the semiclosed container, to prevent oxidation, needs to have available carbon[16] as well as manganese.

The authors have referred to the presence of water vapour in the 'dry' atmospheres necessary for the hydrogen producing $Mn + H_2O$ reaction. The questions therefore arise how wet can the nitrogen be and if industrial nitrogen with a dewpoint of ~ -45°C is acceptable, even possibly

Table 3 Carbon and oxygen contents in Fe–3Mn–0·8C steel compacts independently sintered in semiclosed containers in pure hydrogen atmosphere. NC 100·24 iron powder + Elkem ferromanganese; dewpoint: -60°C

Sintering temperature, °C	Carbon, wt-%	Oxygen, wt-%
1120	0·627	0·316
	0·634	0·303
	0·641	0·311
	(Table 1)	(Table 1)
	0·621	0·244
	0·615	0·257
	0·616	0·248
	0·541	0·178
	0·559	0·196
	0·661	0·208
	0·652	0·181
	0·660	0·215
1150	0·550	0·155
	0·535	0·144
	0·553	0·157
	0·515	0·134
	0·515	0·168
	0·520	0·118
	0·537	0·175
	0·550	0·145
	0·556	0·120
1180	0·533	0·134
	0·528	0·127
	0·483	0·153
	0·505	0·154
	0·483	0·171
	0·476	0·149
	0·522	0·128
	0·504	0·145
	0·525	0·137
1200	0·503	0·118
	0·493	0·124
	0·482	0·112
	0·449	0·126
	0·466	0·119
	0·441	0·107
	0·402	0·125
	0·488	0·108
	0·501	0·116
Green compact: one analysis	0·784	0·229
Nominal composition	0·80	

better. Investigation of the latter question will form the subject of the authors' next communication.[18] The essential carbon need not necessarily be in the form of graphite and positive results are being obtained with hydrocarbon coated starting powders.[19] Another procedure worthy of further attention is providing the necessary manganese and carbon as a getter powder in the semiclosed container. This ensures reducing conditions not only for manganese alloys, but also for steels containing other easily oxidised elements, particularly chromium. Experiments are being carried out with Astaloy CrM with encouraging results.[20]

CONCLUSIONS

1. Provided sintering of a Fe–3Mn–0·6C steel is carried out in a semiclosed container (with availability of carbon and manganese therein, in the present case within the green compact), a furnace atmosphere of dry nitrogen is as effective in preventing formation of deleterious oxide networks as of dry hydrogen, or of H_2–N_2 mixtures.

2. Specimens sintered at 1120 and 1250°C, respectively, possessed similar mechanical properties, irrespective of the H_2–N_2 ratio in the furnace atmosphere.

3. All tensile specimens had strains to failure between 1·6 and 2·0%.

4. For specimens sintered at 1120°C, yield strength $R_{0·2}$ was in the range 386–462 MPa and tensile strength R_m in the range 633–654 MPa.

5. For specimens sintered at 1250°C, $R_{0·2}$ was in the range 426–464 MPa and R_m in the range 724–780 MPa.

ACKNOWLEDGEMENTS

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