Presented at the PM16 International conference on Powder Metallurgy for Automotive and Engineering Industries in Pune, India in 2016. Conference proceedings are available from the Powder Metallurgy Association of India.

Characterization of Iron Powder Mixes Prepared with a Compressible Iron Powder

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Abstract

ATOMET 31 is a compressible iron powder that is produced via iron granulation, grinding and decarburization. This product was specifically developed for the Indian market and is suitable for applications requiring good compressibility and high green strength such as bearing parts, pulleys, oil pump parts and valve guides.

The objective of this paper is to present a thorough description of this product from its production step to the final properties obtained in Fe-C and Fe-Cu-C mixes according to MPIF Standards F-0005, F-0008, and FC-0208. In addition, the effects of different types of graphite on the sintered properties were evaluated. Finally, FC-0208 formulation with 0.45% Fe_3P was included in the study in order to assess its specific effect on the mechanical properties. Microstructures and apparent hardness are provided for various conditions and the results are compared to those achieved with a reference steel powder.

The information provided constitutes a mapping of the properties that can be obtained with this versatile and competitive iron powder.

Introduction

Compressible Fe powder is produced almost exclusively by high pressure water atomization of low carbon steel. To provide a cost effective solution to the Indian PM market, Rio Tinto has developed a specific grade called ATOMET 31. The objective is to anneal it in India at medium term. The originality of this grade is that its production starts from a high carbon liquid iron. This process is specific to Rio Tinto. This paper describes its production process and its properties that are mainly comparable to common water atomized powder but with a higher green strength. As a consequence, ATOMET31 is suitable for a wide range of applications. For the most severe applications, a wide range of Rio Tinto Metal Powders remains available.

Production Route

The production of metal powders at Rio Tinto Metal Powders (RTMP) starts with the smelting of low residual ilmenite ($FeTiO_3$). The resulting co-products of this treatment are titanium dioxide slag (different grades) and high-purity, high carbon liquid iron. Liquid iron is then transferred to the powder plant at the start of the granulation process leading to the production of high quality iron powders for powder metallurgy applications. A flow diagram of the metal powder production route is shown at Fig. 1.

Liquid iron is granulated by pouring into a tundish under which horizontal pressurized water jets disintegrate the flowing stream of liquid iron. During the granulation process, high-carbon iron particles are solidified and partially oxidized. The slurry collected after granulation is dewatered by vacuum and magnetic filtration and further dried in a gas-fired rotary kiln dryer. Granulated powder is then ball-milled to a controlled particle size distribution, suitable for the PM automotive industry. The milled powder is fed to decarburization furnaces operating in the range of 950-1050°C under a reducing hydrogen atmosphere. During annealing, carbon and oxygen from the powder react to form carbon monoxide and dioxide. Hydrogen further helps to deoxidize the metal powder. The annealed product consists in a loosely sintered cake of iron powder. The mechanical action of crusher and disc-mills breaks the powder cake and brings it back to the powder form. Iron powder is then blended as virgin powder or mixed with different additives such as copper, graphite and lubricant to produce consistent premixes for powder metallurgy applications.

ATOMET 31, a compressible iron powder offering high green-strength, is the result of the optimisation of the production process described above.

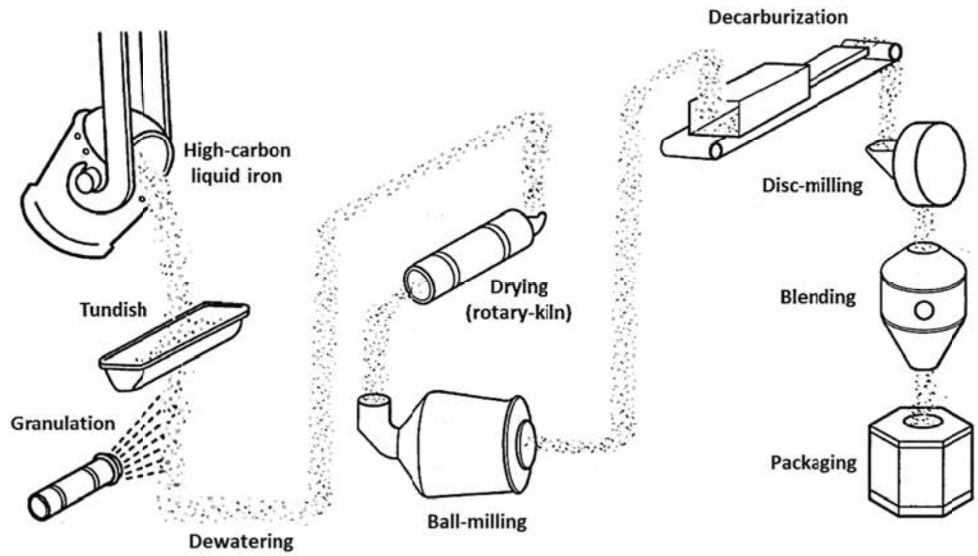

Fig. 1 Process flow diagram for the production of ATOMET 31 [1]

Process Stability

In order to illustrate the production stability of ATOMET 31, the following figures were prepared. Powder characteristics such as %C, %O, apparent density and flow rate are presented for 50 consecutive typical production lots (Fig. 2 and Fig. 3). The typical weight of a single production lot is 36 000 kg. Fig. 4 and 5 show the compaction and sintered properties of standard test mix (MPIF designation FC-0208).

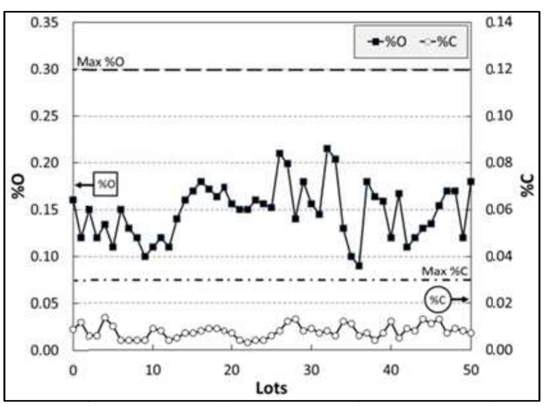

Fig. 2 Carbon and oxygen of 50 consecutive production lots of ATOMET 31

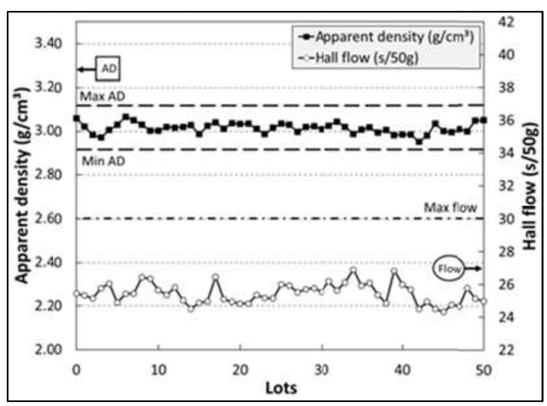

Fig. 3 Apparent density and flow rate (Hall) of 50 consecutive production lots of ATOMET 31

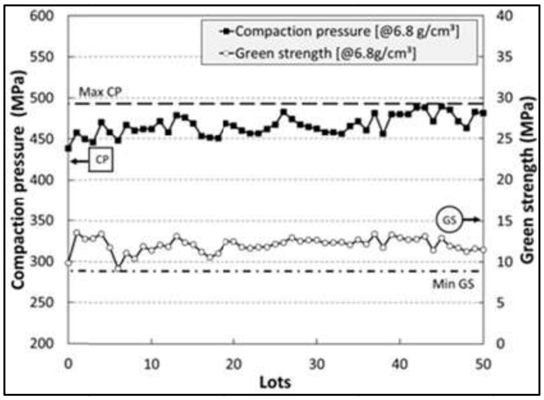

Fig. 4 Compaction pressure and green strength at 6.80 g/cm³ of 50 consecutive production lots of ATOMET 31 (Test mix: Fe - 0.9 C - 2.0 Cu - 0.5 ZnSt)

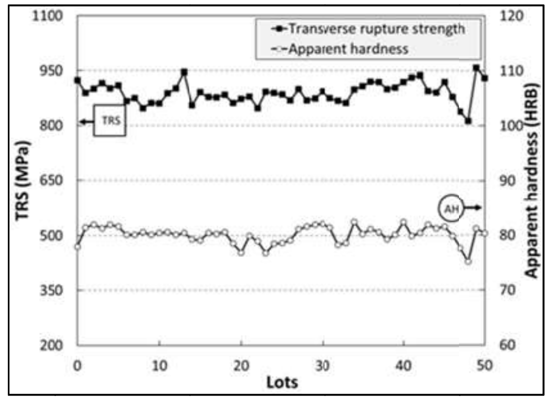

Fig. 5 Transverse rupture strength and apparent hardness of 50 consecutive production lots of ATOMET 31 (Test mix Fe - 0.9 C - 2.0 Cu - 0.5 ZnSt, compacted at 6.80 g/cm³ and sintered at 1121°C for 25 minutes under a 90%N_2/10%H_2 atmosphere)

Table 1 shows the mean values, the minimum and maximum specifications and calculated process capability index (Cpk) for ATOMET 31 and for a standard water-atomized iron powder like ATOMET 1001. It can be seen that in a general manner the lot to lot stability of ATOMET 31 is comparable to ATOMET 1001.

Table 1 : Mean values, specifications and process capability index (Cpk) for ATOMET 31 vs ATOMET 1001

Properties	ATOMET 31				ATOMET 1001			
	Mean	Spec min	Spec max	Cpk*	Mean	Spec min	Spec max	Cpk*
%C	0.008	-	0.03	2.59	0.003	-	0.01	2.04
%O	0.15	-	0.30	1.72	0.08	-	0.15	3.76
Apparent density (g/cm³)	3.02	2.95	3.10	0.92	2.92	2.85	3.00	1.03
Hall flow (s/50g)	25	-	30	2.50	25	-	30	2.51
Green strength at 6.8 g/cm³ (MPa)	12.1	7.9	-	1.55	9.9	7.9	-	0.92
Transverse Rupture Strength (MPa)	890	-	-	-	1048	-	-	-
Apparent hardness (HRB)	80	-	-	-	83	-	-	-

*when there is only the lower limit or upper limit given in the specifications, either Cpl or Cpu is presented.

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Experimental Procedures

This study was performed in two parts. First, the properties of ATOMET 31 were compared to those of ATOMET 1001 and a water atomized competitive iron powder grade. The compressibility and green strength of the powders were compared using mixes containing 0.5% Acrawax. Sintered properties were then measured for three MPIF formulations including F-0005, F-0008, and FC-0208 for which the three iron powders were used. Table 2 summarizes the typical chemical and physical properties of the three base powders. ATOMET 31 shows higher carbon content compared to ATOMET 1001 and the competitive grade. It also contains higher oxygen than ATOMET 1001. The higher carbon and oxygen content in ATOMET 31 can be attributed to the granulation production process. It should be also noted that compared to the other two grades, ATOMET 31 contains much lower percentage of pre-alloyed manganese. The apparent density and flow rate of ATOMET 31 are comparable to the benchmarks.

Table 2 Chemical and physical properties of the three powder grades used in this study

Powder	C, %	O, %	S, %	Mn, %	+150 µm, %	-150/+45 µm, %	-45 µm, %	Apparent density, g/cm³	Flow, s/50 g
ATOMET 31	0.008	0.15	0.0073	0.01	14	67	19	3.02	25
ATOMET 1001	0.002	0.08	0.005	0.21	12	67	21	2.93	25
Comp. G.	0.006	0.19	0.0250	0.18	5	69	22	3.00	25

Table 3 presents the composition of the MPIF grades prepared with the three iron powders. A total of nine mixes were produced. TRS specimens (3.17 x 1.27 x 0.64 cm) were pressed to 6.80 g/cm^3 from each mix. The specimens were then sintered in a mesh belt furnace at 1121°C for 25 minutes in 90%N_2–10%H_2 atmosphere. The dimensional change from die size (DCDS) as well as the transverse rupture strength (TRS) and apparent hardness were evaluated for each mix composition.

Table 3 Composition of mixes prepared with each iron powder

MPIF Grade	Base Fe powder	Copper	Graphite	Lubricant
F-0005	99.4%	-	0.6% PG25*	0.5% Acrawax
F-0008	99.1%	-	0.9% PG25	0.5% Acrawax
FC-0208	97.1%	2%	0.9% PG25	0.5% Acrawax

*Timcal Timrex® PG25 natural graphite, D_{50}=10 µm, D_{90}=22 µm, C%=95.5%.

In the second part of the study, ATOMET 31 was compared only to ATOMET 1001 in FC-0208 formulation. In this part, the effect of phosphorus (added as Fe_3P) and another type of graphite (Asbury 1645) on the properties of the resulting mixes were investigated. The composition of three mixes prepared in the second part is presented in Table 4. From each mix TRS bars and dog-bone specimens were pressed at two densities, 6.80 and 7.10 g/cm^3. Compressibility and green strength were evaluated with TRS bars. TRS bars and dog-bone specimens were sintered at 1121°C for 25 minutes in 90%N_2–10%H_2 atmosphere in a mesh belt furnace. DCDS, TRS and apparent hardness were measured on the TRS bars. Tensile properties including 0.2% offset yield strength, tensile strength, and elongation were measured on dog-bone specimens. Finally, the microstructural analysis was performed on cross section of TRS bars as polished at 100X and after Nital etching at 500X by optical microscopy.

Table 4 Composition of FC-0208 mixes prepared with ATOMET 31 and ATOMET 1001

Mix ID	Base Fe powder	Copper	Graphite	Fe_3P	Lubricant
1	ATOMET 31	2%	0.9% 1645*	-	0.5% Acrawax
2	ATOMET 31	2%	0.9% 1645	0.45%	0.5% Acrawax
3	ATOMET 1001	2%	0.9% 1645	-	0.5% Acrawax

*Asbury 1645 natural graphite, D_{50}=15 µm, D_{90}=37 µm, C%=96.7%.

Results and discussion

The compressibility curve and green strength as a function of green density for ATOMET 31 compared to ATOMET 1001 and the competitive grade are shown in Fig. 6. As shown, ATOMET 31 is less compressible than the ATOMET 1001. This can be explained by the fact that the former is granulated powder whereas the latter is atomized and therefore more compressible. However, ATOMET 31 showed higher compressibility than the competitive grade at lower compaction pressures. At higher compaction pressures, the compressibility of ATOMET 31 was similar to the competitive grade. ATOMET 31 showed higher green strength than the benchmarks at all green densities, the competitive grade had the lowest green strength values.

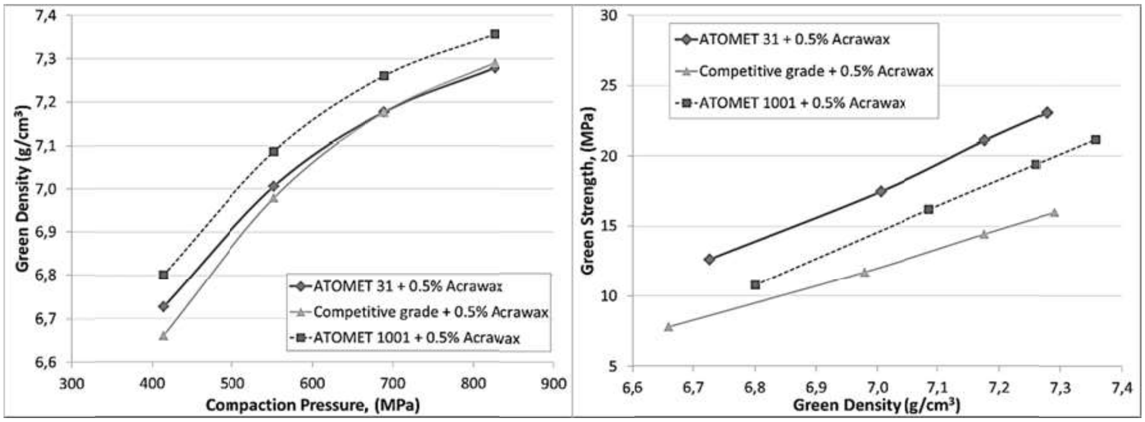


Fig. 6 Compressibility curve and green strength of ATOMET 31 compared to ATOMET 1001 and the competitive grade

The compaction pressure and sintered properties of the TRS bars pressed with different MPIF formulations in the first part of this study are summarized in Table 5.

Table 5 Compaction and sintered properties of TRS bars pressed at 6.80 g/cm^3 density from different mixes containing ATOMET 31, the competitive grade and ATOMET 1001 (standard properties of MPIF grades are presented as reference)

Mix Grade	Base Powder	Compaction pressure (MPa) @ 6.80 g/cm^3	DCDS (%)	TRS (MPa)	Hardness (HRB)
F-0005	ATOMET 31	445	0.06	525	51
	Competitive Grade	462	0.07	578	48
	ATOMET 1001	412	0.08	575	46
	MPIF* @ 6.90 g/cm^3	-	-	520	55
F-0008	ATOMET 31	456	0.11	634	66
	Competitive Grade	462	0.12	714	64
	ATOMET 1001	407	0.13	723	64
	MPIF* @ 6.60 g/cm^3	-	-	510	60
	MPIF* @ 7.00 g/cm^3	-	-	690	70
FC-0208	ATOMET 31	451	0.22	918	78
	Competitive Grade	456	0.31	1042	80
	ATOMET 1001	394	0.30	1038	82
	MPIF* @ 6.70 g/cm^3	-	-	860	73

*The MPIF typical properties reported here do not correspond to the exact densities used in this study

Fig. 7 compares the compressibility of ATOMET 31 with the two benchmarks in different MPIF formulations. At density of 6.8 g/cm^3, all mixes containing ATOMET 31 were more compressible than those with the competitive grade but less compressible than those with ATOMET 1001. This is similar to what was observed on the compressibility of the base powders in Fig. 6. The dimensional change of specimens pressed from mixes with ATOMET 31 and benchmark powders is shown in Fig. 8. In F-

0005 and F-0008, the dimensional change observed for the three different base powders is quite similar. The addition of 2% Cu differentiates substantially the dimensional change of ATOMET 31 which shows less growth than the two other base powders. It is also shown that in FC-0208, the DCDS is significantly higher than that of F-0005 and F-0008 due to the addition of copper. The TRS of ATOMET 31 specimens were lower than those of benchmarks for all the grades tested as shown in Fig. 9. This can be explained by the fact that ATOMET 31 contains lower amounts of pre-alloyed manganese than ATOMET 1001 and the competitive grade. The presence of manganese contributes to the improvement of the mechanical properties by solid solution strengthening. The hardness of ATOMET 31 based grades was similar to the benchmarks (see Fig. 9). Both TRS and hardness increased with the increase of graphite content and with the addition of copper as expected. As shown in Table 5, the mechanical properties of all ATOMET 31 mixes are comparable to or better than those of the typical values reported by MPIF for F-0005, F-0008, and FC-0208 grades [2].

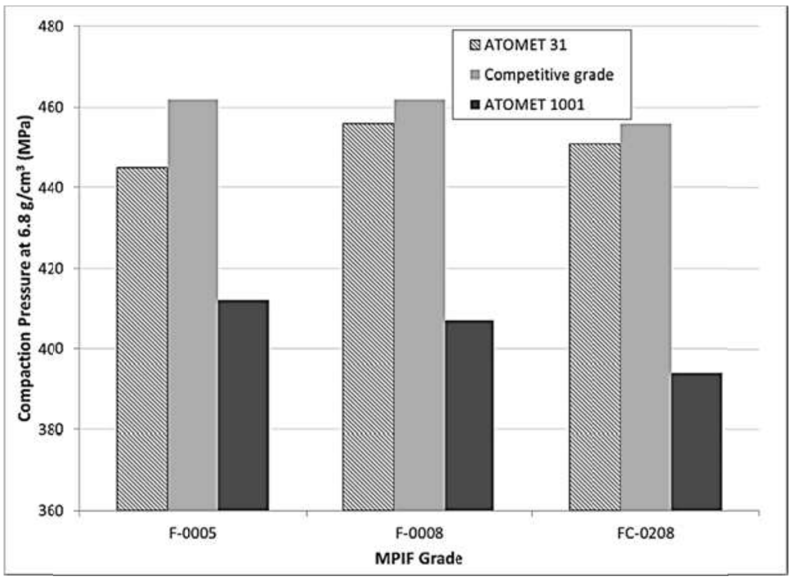


Fig. 7 Compressibility of different mixes based on ATOMET 31, competitive grade and ATOMET 1001 at 6.80 g/cm^3 density

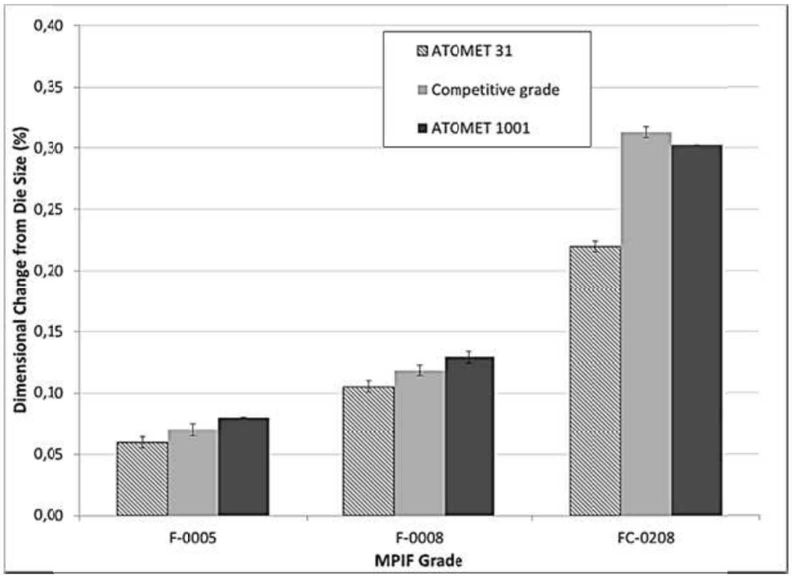


Fig. 8 Dimensional change from die size of TRS bars pressed from different mixes based on ATOMET 31, competitive grade and ATOMET 1001 at 6.80 g/cm^3 density: the error bars presented in this study represent standard deviation (σ)

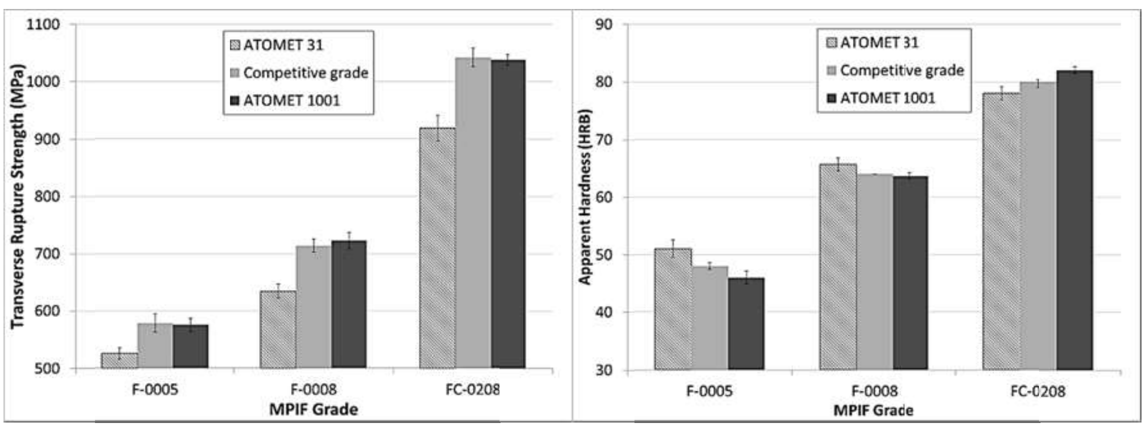


Fig. 9 TRS and hardness of bars pressed from different mixes based on ATOMET 31, competitive grade and ATOMET 1001 at 6.80 g/cm^3 density

In the second part of the study, ATOMET 31 was compared to ATOMET 1001 only in FC-0208 formulation. In this part, the effect of phosphorus addition on the properties of the mix with ATOMET 31 was investigated. Also, Asbury 1645 natural graphite with coarser particle size distribution was used in the preparation of the mixes to investigate its effect specifically on the dimensional change. The results of this part, including green properties, TRS, dimensional change from die size, and tensile properties, are presented in Table 6.

Table 6 Green and sintered properties of specimens pressed at 6.80 and 7.10 g/cm³ density from FC-0208 mixes containing ATOMET 31, ATOMET 31 + Fe_3P and ATOMET 1001; the typical properties of MPIF FC-0208 grade are presented as reference

Base Powder	Compressibility (MPa)	Green Strength (MPa)	DCDS (%)	TRS (MPa)	Hardness (HRB)	Yield Strenght (MPa)	UTS (MPa)	Elongation (%)	Carbon (%)
			@ 6.80 g/cm³						
ATOMET 31	466	13.4	0.26	869	81	445	526	1.13	-
AT. 31 + Fe_3P	467	13.1	0.25	862	81	464	533	0.98	-
ATOMET 1001	408	10.3	0.29	1010	82	465	580	1.65	-
			@ 7.10 g/cm³						
ATOMET 31	725	17.8	0.35	1080	87	501	589	0.82	0.75
AT. 31 + Fe_3P	728	18.3	0.33	1093	89	515	584	0.67	0.73
ATOMET 1001	586	14.0	0.35	1236	91	521	697	1.79	0.77
MPIF* FC-0208 @ 7.20 g/cm³	-	-	-	1070	84	450	520	<1	-

*The MPIF typical properties reported here do not correspond to the exact densities used in this study

As demonstrated in Fig. 10, the ATOMET 31 mix required higher compaction pressure to reach 6.80 and 7.10 g/cm³ density compared to ATOMET 1001. The green strength of ATOMET 31 mix was however significantly higher than that of ATOMET 1001 at both densities. As phosphorus had no effect the on the compressibility and green strength of ATOMET 31, it was not presented in the figure.

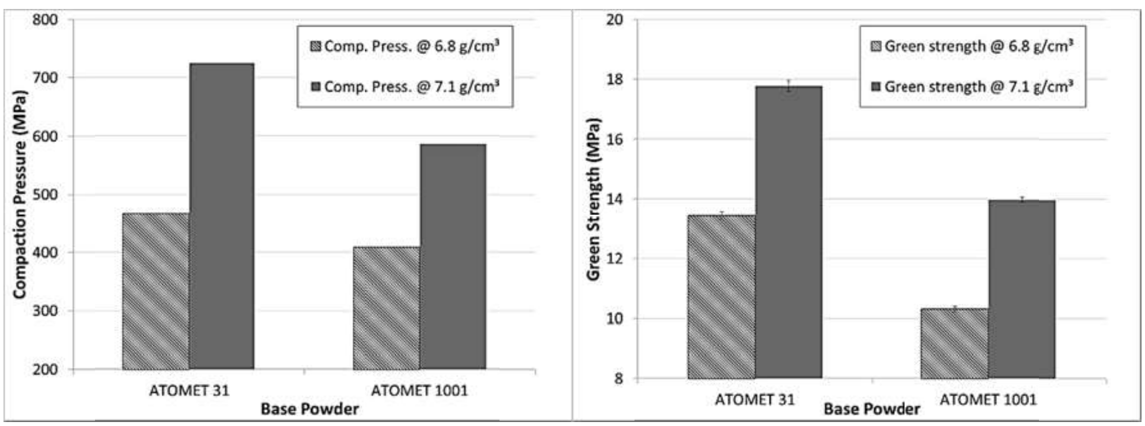


Fig. 10 Compaction pressure and green strength of FC-0208 mixes based on ATOMET 31 and ATOMET 1001 at 6.80 and 7.10 g/cm³ density

As shown in Fig. 11, for the sintered TRS bars, DCDS of the ATOMET 31 mix was 0.03% lower compared to ATOMET 1001 at 6.80 g/cm³ density. For bars pressed at 7.10 g/cm³, dimensional change was similar for ATOMET 31 and ATOMET 1001 mixes. These results on dimensional change are different from those of the first part of study where a large difference in the dimensional change (-0.08%) was observed for bars pressed with the ATOMET 31 mix compared to ATOMET 1001.

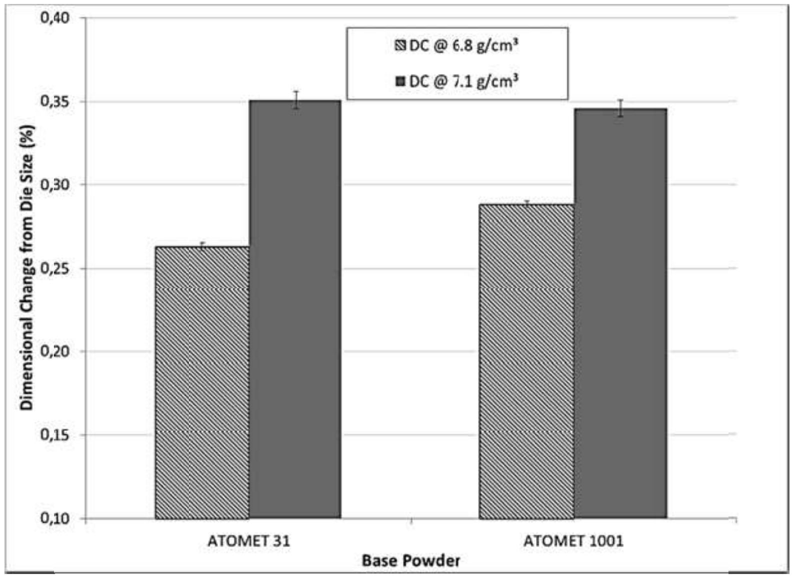


Fig. 11 Dimensional change from die size of sintered TRS bars pressed at 6.80 and 7.10 g/cm^3 density with FC-0208 mixes based on ATOMET 31 and ATOMET 1001

Fig. 12 compares all values of dimensional change of FC-0208 grade obtained in the first and second part of this study for ATOMET 31 and ATOMET 1001. It is interesting to note that the use of graphite type B increased the dimensional change of ATOMET 31 parts pressed at 6.80 g/cm^3 density by 0.04%. Also, compacting at higher density of 7.10 g/cm^3 had more effect on the increase of dimensional change for ATOMET 31 (+0.09%) than ATOMET 1001 (+0.06%). Therefore, the combined effect of type of graphite and density can be used as a solution to increase the dimensional change of ATOMET 31 mixes to the level of ATOMET 1001.

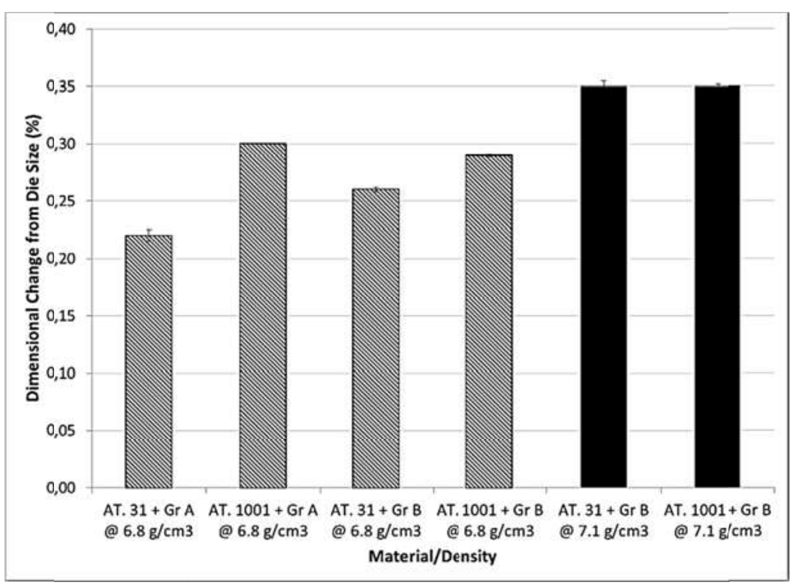


Fig. 12 Dimensional change from die size of sintered TRS bars pressed at 6.80 and 7.10 g/cm^3 density with FC-0208 mixes based on ATOMET 31 and ATOMET 1001: results combined from the first and the second part of study

The TRS and apparent hardness of ATOMET 31 mixes are compared to ATOMET 1001 in Fig. 13. The ATOMET 31 mix showed lower TRS compared to ATOMET 1001 which could be again related to the presence of manganese in the latter. The hardness of ATOMET 31 mix was similar to ATOMET 1001 at 6.80 g/cm^3. At 7.10 g/cm^3 density, hardness of ATOMET 31 was slightly lower than ATOMET 1001.This difference in hardness can also be explained by the slight difference in sintered carbon content of the materials.

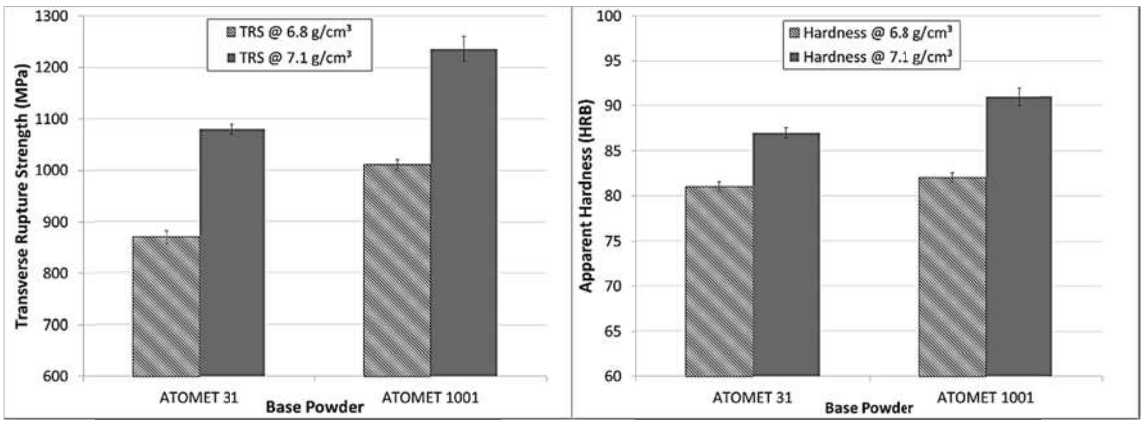


Fig. 13 TRS and apparent hardness of sintered bars pressed at 6.8 and 7.1 g/cm^3 density with FC-0208 mixes based on ATOMET 31 and ATOMET 1001

Fig. 14 compares the yield strength of FC-0208 materials prepared with ATOMET 31, ATOMET 31 + Fe_3P and ATOMET 1001. The yield strength of ATOMET 31 was about 4% lower than that of ATOMET 1001 at both densities. For all materials, yield strength increased by 10% with the increase in density from 6.80 to 7.10 g/cm^3. The average yield strength of ATOMET 31 at 6.80 g/cm^3 seemed to improve by the addition of phosphorus, but considering the variation of yield strength between the specimens, the difference was not significant. However, at higher density of 7.10 g/cm^3, phosphorus showed to improve the yield strength of ATOMET 31. Given the fact that ATOMET 31 is granulated and does not contain manganese, it can be considered as a good low cost alternative to ATOMET 1001 in certain applications as its yield strength was only 4% below that the benchmark. It should be noted that from an application point of view, yield strength is more representative than TRS, since the PM parts do not function in a plastic mode.

The tensile strengths of ATOMET 31 were 526 and 589 MPa at 6.80 and 7.10 g/cm^3 density, respectively. These values were 10 and 15% lower than those of ATOMET 1001, but still within the specifications of FC-0208 grade. As shown in Table 6, the elongation of ATOMET 31 mixes was lower than that of ATOMET 1001 at both densities. The addition of Fe_3P seemed to reduce slightly the elongation of ATOMET 31. This can be possibly explained by the role of phosphorus on the embrittlement of grain boundaries.

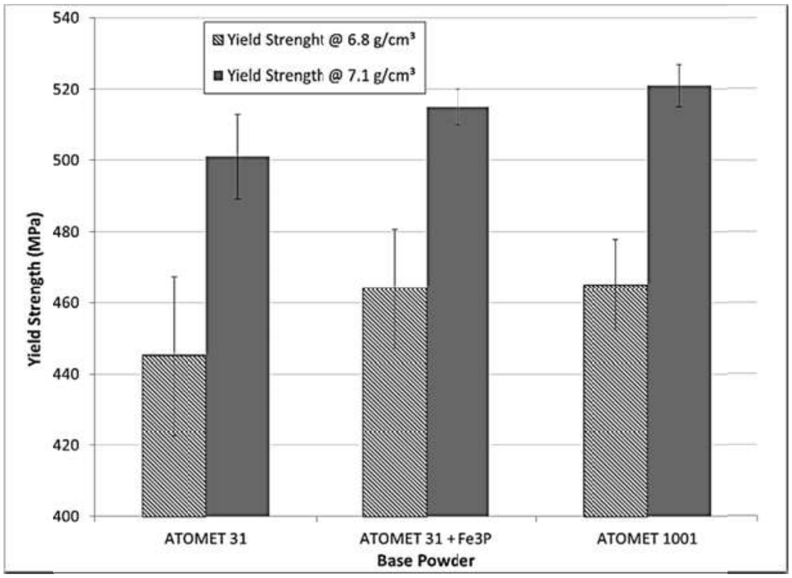


Fig. 14 0.2% offset yield strength of sintered specimens pressed at 6.80 and 7.10 g/cm^3 density with FC-0208 mixes based on ATOMET 31, ATOMET 31 + Fe_3P, and ATOMET 1001

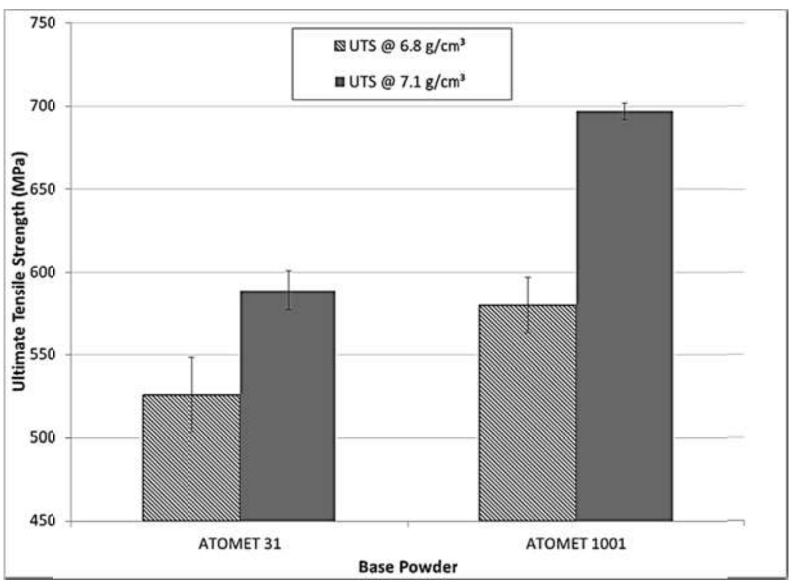
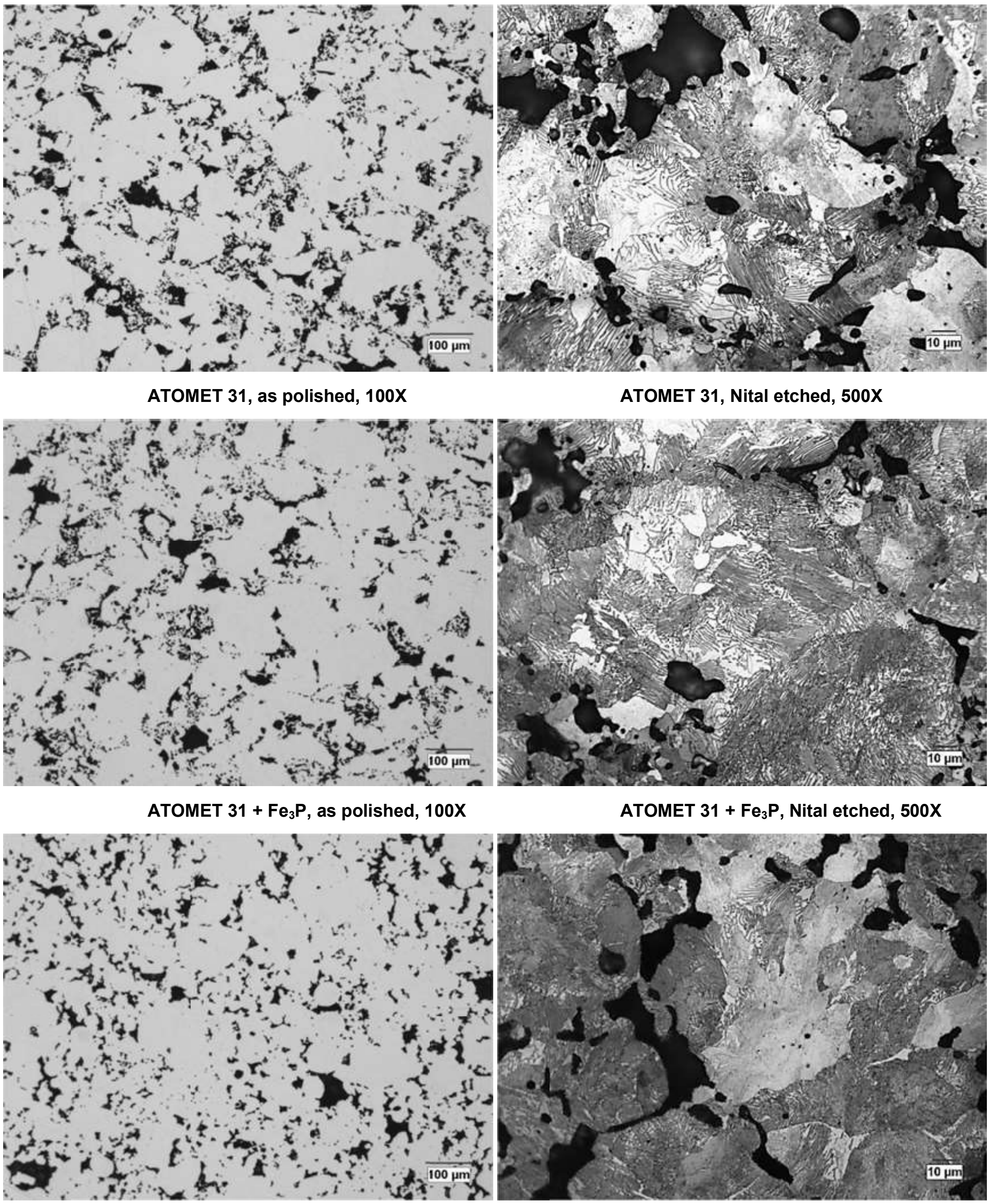


Fig. 15 Ultimate tensile strength of sintered specimens pressed at 6.80 and 7.10 g/cm^3 density with FC-0208 mixes based on ATOMET 31 and ATOMET 1001

The microstructures of FC-0208 TRS bars at 6.80 g/cm^3 density with different iron powders are presented in Fig. 16. The distribution of porosity in ATOMET 31 specimens, as shown in the as polished images, is different from that of ATOMET 1001 since the former is a granulated iron powder and the latter is an atomized iron powder. The etched microstructure of ATOMET 31 is typical of FC-0208 structure and contains pearlite and a small amount of ferrite. In ATOMET 1001, the pearlite phase was clearly finer than in ATOMET 31 due to the presence of manganese. Also, the microstructure of ATOMET 1001 specimens contained almost no ferrite which can be related to their slightly higher carbon content compared to ATOMET 31. These microstructural observations are in line with the superior mechanical properties of ATOMET1001. The effect of phosphorus on the microstructure with ATOMET 31 was not obvious in this study.



ATOMET 31, as polished, 100X | **ATOMET 31, Nital etched, 500X**

ATOMET 31 + Fe_3P, as polished, 100X | **ATOMET 31 + Fe_3P, Nital etched, 500X**

ATOMET 1001, as polished, 100X | **ATOMET 1001, Nital etched, 500X**

Fig. 16 Microstructure of sintered TRS bars at 6.80 g/cm^3 density from FC-0208 mixes with different base iron powders

Conclusion

ATOMET 31 shows compressibility comparable to the competitive grade used in this study and higher green strength than both benchmarks including ATOMET 1001. For MPIF FC-0208 formulation, the combined effect of type of graphite and density can be used as a solution to increase the dimensional change of ATOMET 31 mixes to the level of ATOMET 1001. The yield strength of ATOMET 31 mix based on FC-0208 grade at 7.10 g/cm^3 was similar to that of ATOMET 1001. Given the fact that ATOMET 31 is granulated and does not contain manganese, it can be considered as a good low cost alternative to ATOMET 1001 in certain applications. Also, the addition of phosphorus to ATOMET 31 mixes can be used as a means to improve yield strength.

Reference

[1] M.L. Marucci, “Production of Powder Metallurgy Carbon and Low-Alloy Steels”: Powder Metallurgy, ASM Handbook, Volume 7, ASM International, Edition 2015.

[2] MPIF Standard 35, “Materials Standards for PM Structural Parts”, Edition 2012.