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Pressed and sintered AISI 4140 PM low alloy steel from gas atomised powders

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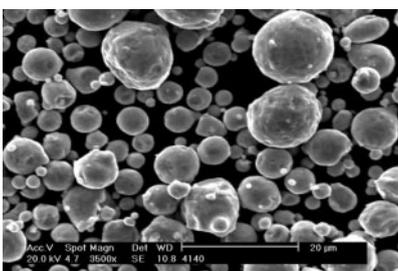
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In conventional PM of low alloy steels various alloying routes are used (fully prealloyed powders, diffusion alloying, elemental powders), but always using powders that allow uniaxial pressing, i.e. acceptable compressibility and flow. Fully prealloyed gas atomised powders (including carbon content) have never been an option because their small size. These powders need to be granulated before being uniaxially pressed and the binder used in the granulating process must be eliminated in the first steps of the sintering cycle. Such a processing route is proposed and initial results presented. A potential advantage of the process is that a low particle size can activate the sintering performance, bringing energy and cost savings over the full process cycle.

The chemical compositions of PM low alloy steels are usually far from those of conventional wrought steels. Elements such as Cu or P (normally avoided in conventional steels) have always been used; in the late 1990s Ni and Mo became more frequent, and more recently, elements with high oxygen affinity like Cr or Mn have gained importance.¹ This situation has promoted specific PM standards regarding chemical compositions because the total alloying content does not fit the concept of conventional ingot metallurgy 'low alloy steels'. Recently, the concept of 'lean steels' has stimulated some PM compositions similar to those of wrought low alloy steels. In contrast, low alloy steels under conventional standards (in the sense of the chemical composition in Table 1), e.g. AISI 4140, have been used in metal injection moulding (MIM). In MIM, gas atomised powders (with less possibility to capture oxygen) and more controlled atmospheres during sintering (owing to the use of batch furnaces) have allowed the use of these standard grades without any problem. In the conventional press and sinter process these grades have never been used, and the powder produced for MIM applications is not suitable for uniaxial compaction.



1 SEM image of AISI 4140 gas atomised powder

In the present work an approach has been made to develop pressed and sintered steels using an AISI 4140 powder developed for MIM, and thereby to profit from the small size of the particles in the level of sintering activation achieved.²

Experimental process

The powder used is an AISI 4140 grade gas atomised with an average particle size of 22 µm (by Sandvik Osprey Ltd, UK). The chemical composition and size distribution of the powder are summarised in Tables 1 and 2 and the morphology of the powder is shown in Fig. 1.

To facilitate uniaxial pressing, the powder was granulated following the established internal protocol of Abrasivos Grinding SA for manufacturing diamond tooling. The binder is a thermosetting resin that is fully decomposed at 400°C. Granules of 0.5 mm average diameter were obtained with good compressibility and flow characteristics. Before the granulating process, the MIM powder was mixed in a Turbula mixer for 20 min to assure, after sintering, a carbon content of 0.7%. LECO analysis after sintering gave a measured carbon content of 0.9 wt-%.

The granules obtained were die pressed at 700 MPa. They were then submitted to a combined cycle of debinding and sintering. Green samples

were heated to 400°C for 30 min then heated to the sintering temperature. Sintering was carried out in 90%N₂-10%H₂-0.1%CH₄ at 1200 or 1300°C for 30 min. Specimens were then cooled in the furnace at low cooling rate. Sintered density, hardness, tensile features and microstructure were evaluated.

Results

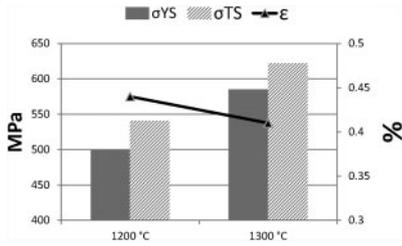
Density and hardness results are summarised in Table 3. Density reached lower values than expected from the particle size. The hardness was slightly higher than expected for this density and composition of the steel, but is in accordance with the microstructure obtained (see below).

The tensile properties are summarised in Fig. 2. The values for UTS and yield strength were comparable with high standard PM grades, but the elongation was rather poor.

The microstructures obtained are shown in Fig. 3. In the upper part of the figure the microstructural constituents are identified, on the basis of microhardness measures. Even though the cooling rate was extremely low, non-equilibrium microstructures can be found in both specimens. In the

Table 1 Chemical analysis of experimental AISI 4140 powder³/wt-%

C	Si	Mn	P	S	Cr	Mo
0.38-0.43	0.15-0.35	0.75-1.00	<0.035	<0.04	0.8-1.1	0.15-0.25



2 Ultimate tensile strength, yield strength and elongation of steels sintered at 1200 and 1300°C

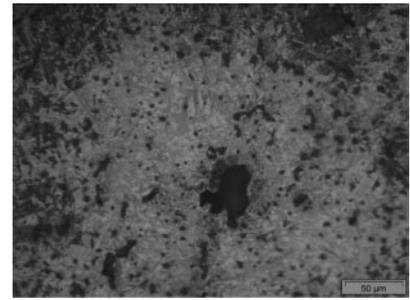
material sintered at 1200°C a combination of bainite (upper and lower) and martensite was found, whereas at 1300°C, mostly lower bainite and martensite were observed. At 1300°C some secondary porosity can be found across the microstructure, which is typical of transient liquid phase sintering. It is possible that very low segregations of the alloying elements (especially Mn and Mo⁴) could promote a transient liquid phase and, as a consequence, enrichment of the surrounding area in alloying elements and therefore a localised increase in the hardenability.⁵ This is the cause of the large amount of martensite surrounding such secondary porosity. Semi-quantitative SEM-EDAX analysis revealed no evidence of this

Table 2 Size distribution of AISI 4140 powder³

	$D_{90}/\mu\text{m}$	$D_{50}/\mu\text{m}$	$D_{10}/\mu\text{m}$
80%	22	27.0	11.5
90%	22	22.0	10.5
90%	16	16.0	9.0

possible segregation; differential thermal analysis was also performed to attempt to detect transient melting between 1200 and 1300°C, but without success. However, work in the literature on steels with high Mn contents under similar sintering conditions have also found this secondary porosity with the same microstructural effect.⁴ A detailed view of such a phenomenon is shown in Fig. 4.

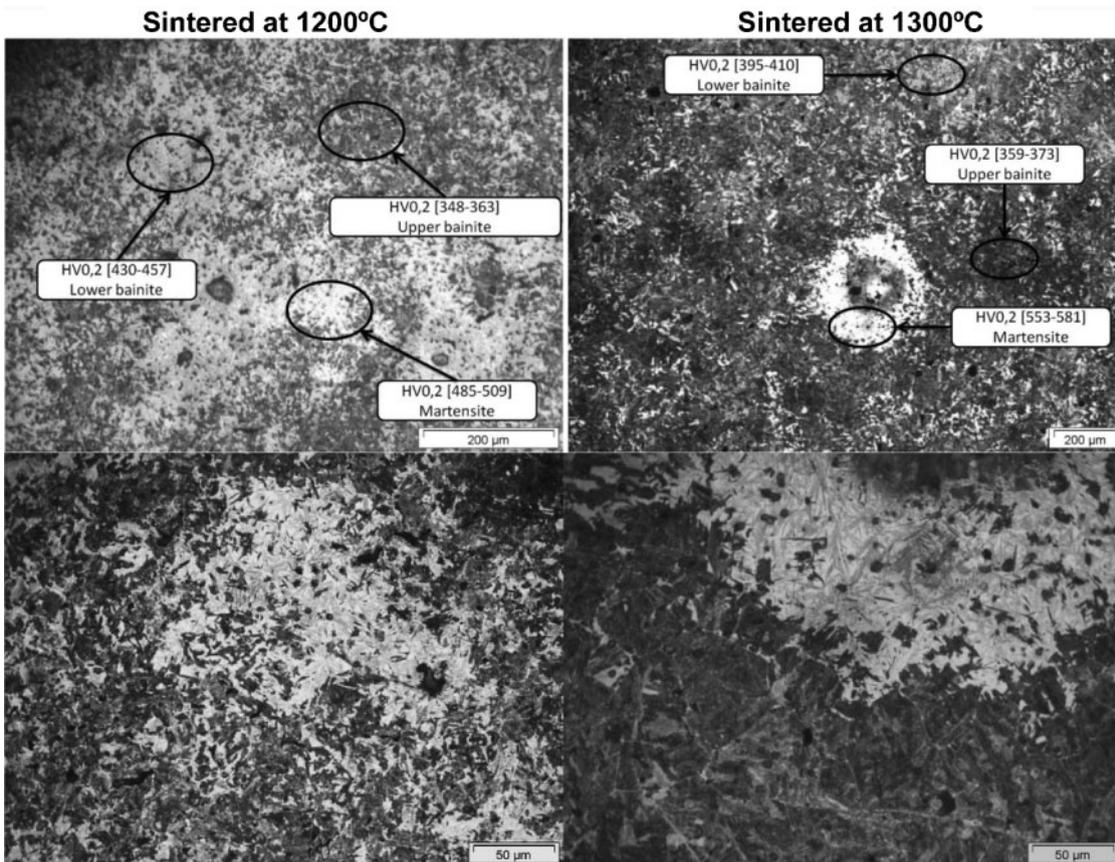
The fracture behaviour for this steels is brittle (consistent with the poor elongation values and the narrow range between the yield strength and the tensile strength). The microfracture behaviour is also brittle, as can be seen in Fig. 5. In all cases, most of the fracture surface is composed of cleavage facets. The obtained properties are fully consistent with the observed microstructure.



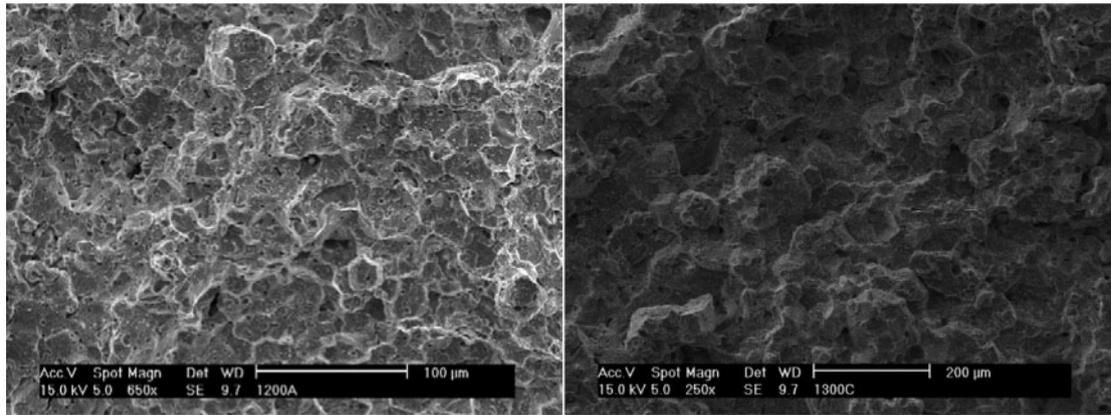
4 Detail of material sintered at 1300°C

Discussion

The composition of the gas atomised powder, which is fully prealloyed, gives a good distribution of the alloying elements that promotes, even at low cooling rates, the transformation of austenite to lower bainite and martensite. At higher sintering temperature, any possible microsegregation disappears, allowing higher amounts of hard phases after cooling. The presence of these microconstituents is responsible for the higher hardness and tensile strength, as well as the lower elongation. The fracture surfaces obtained suggest good sintering, in that the load bearing cross-section is high. The mechanical



3 Microstructure of sintered steels (nital etch): microhardness of identified micro-constituents shown in upper images



5 Tensile fracture surfaces of steels sintered at 1200°C (left) and 1300°C (right)

Table 3 Density and hardness of sintered steel

Sintering temp./°C	Density/g cm ⁻³	Hardness/HV30
1200	7.1	328 ± 18
1300	7.2	329 ± 14

properties of the present materials compare well with high chromium PM steels (which have higher amount of alloying elements).⁶

The developed process is fully adapted to conventional routes for manufacturing low alloy PM steels and uses a powder with much lower alloying content than 'high performance' PM steels.⁷ The present steels are heat treatable, thus all of their properties (particularly elongation) can be improved by an appropriate heat treatment.

Conclusions

In this paper, an alternative route to produce low alloy PM steels has been developed. The main difference from conventional routes is the use of fine spherical powders that cannot be pressed uniaxially. This type of powder allows similar levels of performance to be obtained to conventional grades with much higher amount of alloying elements. Although the result reported above are preliminary, the alternative process shows distinct promise and the full economical advantages should be industrially evaluated.

Acknowledgement

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