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Processing and properties of sinter hardened Fe–Cr–Mo steels with admixed extra-fine nickel

M. Gauthier^{*1}, S. Metcalfe², S. Pelletier¹ and T. F. Stephenson²

The processing and properties of chromium–molybdenum, powder metallurgy steels with admixed extra-fine nickel (XF Ni) were investigated. Prealloyed Fe–1.5Cr–0.2Mo powder was blended with different quantities of XF Ni, while a hybrid steel with lower Cr content was prepared by blending Fe–1.5Cr–0.2Mo and Fe–0.5Mo prealloyed powders, with additions of XF Ni and copper powders. These steels were compacted into different part shapes in order to evaluate the effect of part thickness on sinterhardening behaviour. These parts were also subjected to different cooling rates after sintering. This study showed that additions of XF Ni improve the compressibility, densification behaviour and mechanical properties of Cr–Mo steels. Furthermore, the properties of the hybrid steel were shown to be either equal to or greater than those of the reference material. Hardenability of all steels was sufficiently high such that part thickness was seen to have negligible impact. Higher cooling rates generally resulted in improved mechanical properties.

Keywords: Chromium–molybdenum steels, Extra-fine nickel, Sinterhardening, Hybrid steels, Mechanical properties, Cooling rate

Introduction

The hardenability of steels can be increased through the use of different alloying elements (e.g. manganese, chromium, molybdenum, nickel, silicon, etc.) combined with a controlled cooling rate after heat treatment.¹ In powder metallurgy (PM) materials, the use of controlled cooling conditions after sintering in the high temperature zone of the furnace can impact the hardenability of steels. During this sinter hardening process, molybdenum, nickel and copper are commonly used to increase the mechanical properties of PM steels, as the oxides of Mo, Ni and Cu are readily reducible under standard sintering conditions.^{2,3} Sinter hardening thus provides a way to obtain hard martensite structures in PM steels without the otherwise essential post-sintering steps of austenitising, oil-quenching and tempering.⁴ Chromium based PM steels are interesting materials with good sinter hardening behaviour for high strength PM applications⁵ and have been shown to be advantageous for fatigue loaded parts.^{6,7} Such Cr-containing PM alloys can readily be processed using modern sintering practices.⁸ The high stability of chromium oxides traditionally required sintering at very high temperatures, which reduced the commercial interest of using the otherwise high hardenability potential of Cr in PM.⁹

High static and dynamic mechanical properties are required for parts such as gears and sprockets. To improve the microstructure of PM steels so that their mechanical properties (e.g. tensile, hardness, wear resistance) can be increased to meet the stringent requirements for these applications, enhancing the hardenability of these steels through careful choice of alloying elements and processing conditions is essential. Using prealloyed powders can help in improving chemical and phase homogeneity,¹⁰ but compressibility suffers from the solution hardening effect of alloyed elements. In this context, the use of admixed extra-fine nickel (XF Ni) instead of standard Ni grades as been shown to improve nickel distribution and mechanical properties of PM steels.^{11,12}

This paper presents the results of an investigation of the impact of XF Ni additions on the processing and static properties of sinter hardened, chromium–molybdenum PM steels. The influence of such additions was also studied in the case of a hybrid steel containing half the Cr content of the reference alloy. As part thickness or weight¹³ and cooling rate (which depends on the furnace cooling conditions and part density/porosity)^{14,15} are important factors for sinterhardening of PM steels, they were also varied (at three levels each) and their influence on sinterhardened steel properties was determined.

Some of the results reported in this paper were previously presented in a conference paper by the same authors.¹⁶

Experimental

Four different powder formulations were prepared as given in Table 1. In order to prepare the first three powder

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blends, the chosen prealloyed Fe–Cr–Mo powder was Astaloy CrL (Höganäs), which contains 1.5 wt-%Cr and 0.2 wt-%Mo. Additions of 0.65 wt-% graphite (Asbury SW 1651), 0.75 wt-% Acrawax C (Lonza) lubricant and either 0, 1 or 2 wt-% of extra-fine nickel T110D (Vale Inco) were made to this base powder. The hybrid steel was obtained by using a 50/50 (by weight) blend of Astaloy CrL and prealloyed Fe–0.5 wt-%Mo ATOMET 4001 (Rio Tinto Metal Powders), for total Cr and Mo contents of 0.75 and 0.35 wt-% respectively. The same types and amounts of graphite and lubricant were used, but 0.7 wt-% ACuPowder 301 copper powder (ACuPowder) was added, as well as 1 wt-%Ni T110D. Total combined carbon content was confirmed to be ~ 0.6 wt-% for every series after sinter hardening. A twin shell V-blender was used to prepare a 68 kg batch of each of the four powder blends.

A Gasbarre, fully instrumented, 150-ton mechanical press was used to compact these blends into parts of three different shapes, namely transverse rupture strength (TRS) bars ($38 \times 12.7 \times 6.35$ mm), IZOD bars ($76.2 \times 12.7 \times 12.7$ mm) and rings (50.8 mm OD, 19.05 mm ID, and 25.4 mm long), as shown in Fig. 1. The parts were produced to a fixed, target green density of 7.00 ± 0.02 g cm $^{-3}$. An Abbott five-zone, 30 cm wide hybrid pusher furnace with a Varicool Rapid Cooling Unit was used to sinter selected specimens of each part type. Sintering was carried out for 30 min at 1140°C in a 10%H $_2$ –90%N $_2$ atmosphere. Three cooling rates were chosen: low ($\sim 0.8^\circ\text{C s}^{-1}$), medium ($\sim 2^\circ\text{C s}^{-1}$) and high ($\sim 3^\circ\text{C s}^{-1}$), each applied to a third of the total number of specimens of each type. All the samples were subsequently tempered in air for 1 h at 180–200°C. The average, final weight of the different parts was 18, 83 and 335 g for the TRS, IZOD and rings respectively.

The sintered density of selected samples for each mix, shape and cooling rate was evaluated, while the dimensional change from die size (ASTM B 610) was measured only on selected TRS bars. These bars were also used to evaluate three-point transverse rupture strength according to MPIF standard 41. In order to evaluate the hardenability of the different steels and the influence of part shape and cooling rate, microindentation hardness profiles (MPIF standard 43) were measured through the smallest section of TRS bars (6.35 mm), IZOD bars (12.7 mm) and rings (15.9 mm). Microindentation values were obtained in Vickers units and converted to Rockwell A (HRA) using tables provided in ASTM Standard E140–97. Five IZOD bars for each mix and cooling rate were machined by grinding for tensile testing according to the ASTM E8-01 standard and their machined density was measured. Tensile tests were conducted on machined tensile specimens with a gauge length of 25.4 mm and a nominal diameter of 5.08 mm at the ends of the gauge section and 4.95 mm at the centre. This slightly lower diameter than the 6.35 mm recommended in ASTM E8-01 was chosen in order to ensure breakage in the gauge section. Tensile testing was performed on an Instron

1 Part shapes

2 Compressibility data as function of formulation for IZOD bars

testing machine at a crosshead speed of 0.635 mm min $^{-1}$. Elongation values were obtained using an extensometer.

Nickel (or nickel and copper) dispersion in sinter hardened ring samples was evaluated on polished cross-sections by X-ray mapping using an Hitachi S-4700 field emission gun scanning electron microscope.

Microstructural evaluation was performed on etched (2% nital) cross-sections of ring specimens and the results of the metallographic analysis were presented in Gauthier *et al.*,¹⁶ and will only be summarised in the current paper.

Results

Compressibility and green density

Compressibility curves were not specifically determined during this study. However, limited compressibility data could be obtained from the compaction operation. The results for the IZOD bars are presented in Fig. 2. As can be seen from this set of data, the compressibility of the powder mixes increased with nickel content, while the hybrid blend containing nickel and copper was the most compressible formulation. In order to reach the target green density of 7.00 g cm $^{-3}$, the reference alloy requires a compressive stress within a range of about 590–

Table 1 Powder formulations

Formulation ↓	Fe/wt-%	Cr/wt-%	Mo/wt-%	Cu/wt-%	C/wt-%	Lubricant/wt-%	XF Ni/wt-%
CrL + 0Ni	Bal.	1.5	0.2	...	0.65	0.75	0
CrL + 1Ni	Bal.	1.5	0.2	...	0.65	0.75	1
CrL + 2Ni	Bal.	1.5	0.2	...	0.65	0.75	2
Hybrid + Ni + Cu	Bal.	0.75	0.35	0.7	0.65	0.75	1

3 Average green density for TRS bars, IZOD bars and rings

620 MPa. This compaction pressure is comparable to the result obtained by Actis Grande *et al.* for a similar alloy,¹⁷ as they needed 600 MPa to reach a 7.00 g cm^{-3} green density. The 1% XF Ni alloy must be compressed at about 585–600 MPa, while the 2%Ni material reached the density target using a compaction pressure range of approximately 570–585 MPa. The hybrid alloy was clearly more compressible as it needed to be compressed at around 540 MPa to reach the required density.

The average green densities by sample type are shown in Fig. 3. The green densities of all samples were within the targeted $6.98\text{--}7.02 \text{ g cm}^{-3}$ range.

Sintered density

The sintered densities of the specimens as a function of part shape, powder blend and cooling rate are presented in Fig. 4a–c. Each bar in the charts for the IZOD and ring specimens (Fig. 4b and c) gives the average sintered density for five samples, the error bars showing the standard deviation range. In the case of the TRS bars (Fig. 4a), only a single sample was used to measure the sintered density, therefore no error bars are indicated. In these graphs, the horizontal, bold dashed line shows the target green density of 7.00 g cm^{-3} .

These results indicate that densification occurred upon sintering for all sample shapes, although it was less pronounced for the 0%Ni IZOD bars (Fig. 4b versus Fig. 3). The addition of XF Ni is seen to increase sintered density by about the same amount for TRS and IZOD bars, while the rings had the highest sintered densities, reaching about 7.13 g cm^{-3} for 2% of added nickel. When compared to the 0%Ni standard alloy ($\sim 7.04 \text{ g cm}^{-3}$), this represents an increase of close to 0.1 g cm^{-3} . The sintered density of the hybrid alloy is also generally slightly higher than that of the reference. On the other hand, the cooling rate did not seem to have a clear impact on sintered density for the range of alloys and part shapes in this study.

Dimensional change

The results of dimensional change evaluation from die-size are presented in Fig. 5. This graph shows that, while the length of the TRS bars made from the reference CrL alloy swelled, the presence of either 1 or 2% extra-fine nickel caused the bars to shrink with sintering. The bars made from the hybrid alloy also shrunk, but to a lesser extent than the other Ni-containing bars. These dimensional changes can be explained by the fact that Ni additions cause shrinkage and copper additions cause swelling in PM

(a)

(b)

(c)

a TRS bars; b IZOD bars; c rings

4 Density of sinter hardened specimens

steels.¹⁸ It is also seen that an increased cooling rate tends to induce less swelling in 0%Ni alloy bars and more shrinkage in 1 and 2%Ni alloy specimens. The shrinkage of the TRS bars made from the hybrid alloy was not affected by cooling rate. The increasing shrinkage of the non-hybrid, XFNi-containing alloys with higher cooling rates is hard to explain. In fact, as higher cooling rates generally result in an increased proportion of martensite (and lower quantities of retained austenite) and as martensite is less dense than austenite, one would expect to observe less shrinkage for higher cooling rates.¹⁹ Further analyses would be necessary to explain these dimensional changes.

Machined density of tensile specimens

The density of the tensile specimens machined from IZOD bars was measured in oil and is presented in

5 Dimensional change of TRS bars from die-size

6 Machined density of tensile specimens

Fig. 6. The density of the machined tensile specimens was lower than that of the IZOD bars (Fig. 4b). This was expected as the denser, outer shell of the IZOD bars was removed by the machining operation. Much material was removed as the 12.7 × 12.7 mm square cross-section was machined to a circular, 5.08 mm diameter (4.95 mm in the central portion) cross-section. The density drop was close to about 0.03 g cm⁻³ in all cases, so the general tendencies observed for sintered density versus nickel content and cooling rate for IZOD bars still hold for the machined tensile specimens.

Apparent surface hardness

As detailed in Gauthier *et al.*,¹⁶ microindentation hardness profiles were measured through the thinnest section of each of the three part shapes, i.e. 6.35 mm (TRS), 12.7 mm (IZOD) and 15.9 mm (rings). These profiles revealed that, under the sinter hardening conditions used in this study, all of the samples, whatever their thickness, had through hardened. Apparent hardness measurements were performed on the surface of the sinter hardened materials and the results are given in Fig. 7a–c.

The influence of part shape was small and expected from the results of surface hardness measurements. The hardness values of the IZOD bars (Fig. 7b) were generally higher than those of the rings (Fig. 7a), which in turn were slightly higher than those of the TRS bars (Fig. 7c). No clear explanation for these observations could be found.

(a)

(b)

(c)

a TRS bars; b IZOD bars; c rings

7 Surface hardness

The hardness of the specimens generally increased with both XF Ni content and cooling rate. Increasing the nickel content from 0 to 2% typically brings a 6 to 14 HRA increase in hardness depending on cooling rate, the larger differences being observed for medium and high cooling rate samples. In the case of the hybrid alloy, the hardness difference compared to the reference is typically between 1 and 5 HRA, with a similar tendency of greater differences at medium and high cooling rates.

The impact of cooling rate was readily apparent, with increased hardness values at higher cooling rates for a given material composition. In the case of IZOD bars and rings, the Ni-containing alloys are generally more responsive to cooling rate than the nickel-free material

a yield; b UTS; c elongation; d Young's modulus

8 Tensile properties of sinter hardened specimens

(Fig. 7b and c). This was not observed for the TRS bars (Fig. 7a) where the surface hardness of the reference (0%Ni) steel had a similar response to cooling rate as the Ni-containing alloys due to the lower mass and reduced need for alloy content to achieve a given hardenability.

Tensile properties

The relevant tensile properties of the specimens are given in Fig. 8a–d. The yield stress values (Fig. 8a) of

the reference series were ~600 MPa and, while the standard deviation intervals preclude any firm appraisal of the effect of the cooling rate, there was a general tendency towards higher average yield stresses at higher cooling rates. Higher Ni contents improved the yield stress, and the cooling rate had a more pronounced impact for 1 and 2% nickel alloys when compared to the reference. As an illustration of this fact, the reference alloy (0%Ni) processed at low cooling rate

a 0%Ni – low cooling rate; b 2%Ni – high cooling rate; c hybrid – high cooling rate

9 Microstructures of etched ring specimens

(a)

(b)

a apparent hardness; b UTS

10 Effect of Ni type on properties taken from Wu *et al.*²¹ and from present work

had an average yield stress of ~ 600 MPa, while admixing 2% nickel and using a high cooling rate increased the yield to 970 MPa. This represents a 60% improvement on yield stress. In the case of the hybrid alloy, the low cooling rate yield stress was similar to that of the reference at the same cooling rate, while an increase in cooling rate was more effective in improving the yield stress values in the case of the diluted alloy than for the reference (e.g. a 20% improvement on yield stress was observed for the hybrid versus the reference at high cooling rates).

The ultimate tensile stress (UTS) (Fig. 8b) showed a similar improvement with increasing nickel content and cooling rate as the yield stress. Admixing 2%Ni and using a high cooling rate result in an average UTS of 1240 MPa, while the reference alloy at low cooling rate only has an UTS of 730 MPa (i.e. a 70% improvement with higher Ni content and higher cooling rate). The UTS of the hybrid alloy was also more sensitive to cooling rate than the reference.

The average elongation values (Fig. 8c) for the reference series were $\sim 2.5\%$. The behaviour of elongation with Ni level and cooling rate (with or without dilution) cannot be precisely defined as the standard deviation values are too large. However, it can tentatively be said that average elongation improved when admixing 1 or 2%Ni at low cooling rate and that medium or high cooling rates for the same Ni levels resulted in elongations lower or equal to those of the reference alloy.

The average Young's Modulus (Fig. 8d) of the reference alloy was ~ 160 GPa. Even though the standard deviations are once again too large to have a clear picture of the effect of composition and cooling rate, the Young's moduli of the other alloys were equal to or less than those of the reference.

Microstructure

Metallographic analysis was carried out for the ring shaped sinter hardened parts. Table 2 summarises the

Table 2 Summary of metallographic analysis results for sinter hardened rings

Alloy type →					
Cooling rate ↓	0%Ni	1%Ni	2%Ni	Hybrid	
Low	Pearlite	Pearlite/martensite	Martensite/pearlite	Martensite/bainite/pearlite	
Medium	Pearlite	Martensite/pearlite	Martensite	Martensite/bainite	
High	Pearlite/ferrite/bainite	Martensite	Martensite	Martensite/bainite	

Table 3 Comparison of Wu *et al.*'s²¹ and the present experimental conditions and results

Source	Ni type	Ni/wt-%	Combined carbon/wt-%	Cu/wt-%	Cooling rate/ °C s ⁻¹	Sintered density (hardness samples)/ g cm ⁻³	Apparent Hardness/ HRA	Sintered density (UTS samples)/ g cm ⁻³	UTS/ MPa
Wu <i>et al.</i> ²¹	Ni123	4	0.65	...	~ 0.1	7.19	59	7.19	901
Present work	Extra fine	0	0.6	...	~ 0.8	7.03	53 ± 2	6.97	734 ± 5
Present work	Extra fine	1	0.6	...	~ 0.8	7.07	58 ± 1	7.04	973 ± 52
Present work	Extra fine	2	0.6	...	~ 0.8	7.08	61 ± 1	7.08	1158 ± 48
Present work (hybrid)	Extra fine	1	0.6	0.7	~ 0.8	7.04	56 ± 1	7.04	767 ± 58

results obtained from this analysis. As was found using X-ray mapping, no nickel or copper-rich areas were identified on any of the specimens. Furthermore, observations at the edges and central areas of the etched samples have confirmed that all of the specimens had through hardened, as was deduced from the micro-indentation hardness measurements.

The microstructure of the reference alloy consisted of pearlite at low (Fig. 9a) and medium cooling rates, while some ferrite and bainite was also observed along with pearlite for high cooling rates. The admixing of 1% XF Ni resulted in the appearance of small amounts of martensite at low cooling rate, which increased in volume at medium cooling rate until the microstructure became fully martensitic at high cooling rates. The material containing 2% XF Ni was predominantly martensitic with some pearlitic areas at low cooling rate, while medium or high cooling rates resulted in a fully martensitic microstructure (Fig. 9b). The hybrid alloy presented martensite and bainite with residual pearlite for low cooling rates. This changed to martensite with some bainite areas for medium and high cooling rates (Fig. 9c).

Discussion

The superior sinterhardenability of Fe–Cr–Mo steels with admixed XF Ni when compared to that of the Ni-free material was demonstrated in this study. In addition, the properties of a sinter hardened hybrid steel with only 0.75% chromium and some XF Ni and Cu additions were shown to be equal or higher than those of the reference alloy. The properties improved at least in part due to the homogeneous microstructures and increased diffusion of XF Ni.

While the performance of this hybrid was generally lower than that of the XF Ni added steels, it must be remembered that these materials were all compacted to the same green density (7.00 g cm^{-3}). Had the powder blends been compacted at the same pressure, the higher compressibility of the hybrid alloy blend would undoubtedly have resulted in higher sintered densities, with a corresponding improvement in mechanical properties. Similarly, a comparison of these alloys at equal sintered densities would probably have reduced the difference between the properties of the hybrid and those of the XF Ni-containing steels.

The results obtained during this investigation can favourably be compared to data from the literature. Indeed, the hardness, TRS, UTS and yield strength are all comparable to the values obtained by Engström *et al.*²⁰ for Ni-free Astaloy CrLbased steels with a slightly higher combined carbon level of 0.75 wt-%C and a range of sintered densities from 6.70 to 7.30 g cm^{-3} (as opposed to 0.60 wt-% combined C and a sintered density of $6.97 \pm 0.01 \text{ g cm}^{-3}$ for the Ni-free alloy of the present work). The results presented in the current paper are therefore representative of the expected mechanical performance of Fe–Cr–Mo steels based on Astaloy CrL.

The effect of using XF Ni instead of standard-sized nickel powder can be illustrated by comparing the present work with that of Wu *et al.*²¹ Indeed, these investigators used standard, Vale Inco type 123 nickel powder²² during their work on the impact of Cr and Mo on the distribution of nickel and carbon in sintered

steels. Figure 10a and b presents a comparison of apparent hardness and UTS values for low cooling rates taken from the present work and Wu *et al.*'s data, while Table 3 compares the main characteristics of these sets of data. In Fig. 10a, the apparent hardness of Wu *et al.*'s Cr–Mo steel made with 4 wt-% standard nickel powder (Std Ni) is 59 HRA while materials produced in the current work with only 2 wt-% XF Ni present an average hardness of about 60 HRA. Furthermore, while the UTS value (Fig. 10b) of the 4 wt-% Std Ni steel is 900 MPa, only 1 wt-% XF Ni was needed to obtain higher mechanical properties (970 MPa), while the UTS reached $\sim 1160 \text{ MPa}$ for 2 wt-% admixed XF Ni. These are interesting results, but considering the fact that the combined carbon level and sintered density of the 4 wt-% Std Ni material were higher than those of the steels made with XF Ni (see Table 3), the difference in mechanical properties could be expected to be even larger if the materials were compared at equal combined carbon and sintered density.

Conclusion

The sinterhardenability of Fe–1.5Cr–0.2Mo PM alloys was shown to be significantly improved by XF Ni additions for parts of three different thicknesses. The admixing of T110D extra-fine nickel powder to Astaloy CrL formulations increased the sintered density, hardness and tensile properties of these materials and also improved their responsiveness to cooling rate. A hybrid alloy prepared by adding 1% XF Ni and 0.7%Cu to a diluted alloy (Fe–0.75Cr–0.35Mo) resulted in properties which were equal or superior to the reference alloy, even though the hybrid had half the chromium level of the standard steel. X-ray mappings have revealed that the diffusion of the admixed nickel (or nickel and copper in the case of the hybrid steel) was very good for the chosen sintering conditions and that these elements were homogeneously distributed in the microstructure of the sinter hardened specimens with no nickel or copper-rich areas found in any of the analysed ring samples. Comparing the results obtained in the present work with data from the literature, lower admixed levels of XF Ni were required to obtain equal or superior mechanical properties than similar alloys processed using standard nickel powder.

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