Fatigue Properties of High Strength Cr-alloyed PM Steels Produced by High Temperature Sintering and Sinter-hardening

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ABSTRACT: Steel powder grades pre-alloyed with Cr are suitable for the manufacturing of high performance PM components, since Cr is a cost-efficient alloying element with excellent hardenability. Cr-alloyed PM steel parts are also fully recyclable, and this is increasingly important from a sustainability perspective. High fatigue strength is a key feature for many applications with high demands, such as synchronizer parts in automotive transmissions. This paper presents fatigue properties for two Cr-alloyed powder grades after different sinter-hardening processes. Sintering trials were undertaken in a mesh belt furnace at 1120 °C, as well as a roller hearth high temperature furnace at 1250 °C. In both trials, rapid cooling was applied after sintering to obtain predominately martensitic microstructures. The fatigue strength of the sinter-hardened and tempered materials were evaluated by plane bending tests on standard test bars. Microstructures were identified and correlated with the fatigue performance of the Cr-alloyed materials.

Key words:
PM steel, chromium, sinter-hardening, high temperature sintering, fatigue strength

1. Introduction

It is well established that powder metallurgy (PM) technology offers a cost-efficient manufacturing route for the production of structural steel components. This is especially true for the production of component series with a complex shape. The near net shape feature of a press-sinter process route provides benefits such as fewer process steps, lower energy consumption and higher productivity. Moreover, by utilizing sinter-hardening where rapid cooling is applied directly after sintering, it is feasible to manufacture high strength PM steel components without the need for a separate heat treatment operation. Furnaces for sinter-hardening are commonly of the continuous mesh belt type, with a maximum operating temperature of around 1150 °C. However, there are also semi-continuous high temperature sintering furnaces for sinter-hardening (e.g. of roller hearth type) which can operate at temperatures up to at least 1300 °C [1]. A higher sintering temperature is beneficial for the mechanical properties of PM steel primarily due to pore rounding and the densification effects. Fatigue strength benefits in particular from these effects, since pores act as crack initiation sites during dynamic loading [2, 3]. Hence, a combination of high temperature sintering and a sinter-hardening process is ideally suited for the production of PM steel components for high fatigue applications, such as synchronizer parts in automotive transmissions.

Steel powder grades pre-alloyed with 1.8-3% Cr are cost-efficient materials and highly suitable for sinter-hardening. These materials provide high mechanical performance after conventional sinter-hardening. By applying high temperature sintering, the mechanical properties can be further enhanced to very high levels [4-10]. This paper presents an investigation of the fatigue properties of sinter-hardened, Cr-alloyed PM steels where the test materials are based on two different pre-alloyed powder grades. Sinter-hardening trials have been undertaken in a mesh belt furnace as well as in a roller hearth high temperature furnace. The fatigue properties were evaluated by 4-point plane bending tests on standard test bars.

2. Experimental Procedure

Two water-atomized powder grades pre-alloyed with 1.8% Cr (Astaloy CrA) and 3.0% Cr + 0.5% Mo (Astaloy CrM), respectively, were used as a base in different test mixes suitable for sinter-hardening. These test mixes are 5 kg pre-mixes which were made by blending base powders and additives in a laboratory Nauta mixer. The additives in the mixes are electrolytic Cu powder (Cu-325), carbonyl Ni powder (Ni-123), synthetic graphite powder (C-F10) and lubricant powder Lube HD. The test mix compositions are presented in Table 1. Mixes with two different graphite contents were made for each test mix in order to adjust for different sintering temperatures (more carbon loss is expected at higher temperature).

Table 1. Compositions of test mixes (in wt.%).

Material	Base powder	Cu (%)	Ni (%)	Graphite* (%)	Lube (%)
CrA-1Cu	Astaloy CrA	1.0	-	0.60/0.65	0.50
CrA-2Cu	Astaloy CrA	2.0	-	0.60/0.65	0.50
CrA-1Ni	Astaloy CrA	-	1.0	0.60/0.65	0.50
CrA-2Ni	Astaloy CrA	-	2.0	0.60/0.65	0.50
CrM	Astaloy CrM	-	-	0.50/0.55	0.50

*) The higher graphite contents were used in the mixes for high temperature sintering.

Standard test bars for fatigue testing (ISO 3928) were produced from the test mixes by uniaxial compaction in a 120 ton hydraulic press. All test bars were compacted to a green density of 7.1 g/cm^3, except for the test bars produced from the CrM mixes which were compacted to 7.0 g/cm^3 in green density. Two different sinter-hardening trials with 30 test bars from each material series were performed in accordance with the process parameters shown in Table 2. The first trial (SH-1120) was conducted in a laboratory-size mesh belt sintering furnace, while the second trial (SH-1250) was conducted in a pilot-size roller hearth high temperature sintering furnace. Small additions of hydro-carbons were used in the N_2/H_2 sintering atmospheres, in order to prevent surface decarburization of the test samples. Both furnaces were equipped with rapid cooling units and using the same cooling rate (~5 °C/s) after sintering was applied in the two trials. Tempering of the sinter-hardened test bars was undertaken at 200 °C for 60 minutes in air.

Table 2. Process parameters used in the sinter-hardening trials.

Process	Sintering T (°C)	Time at T (min)	Atmosphere (vol.%)	Cooling rate (°C/s)
SH-1120	1120	20	N_2/H_2 (90/10) + 0.2% CH_4	~5
SH-1250	1250	20	N_2/H_2 (90/10) + 0.06% C_3H_8	~5

Fatigue testing (4-point plane bending) of the sinter-hardened and tempered test bars was conducted using the staircase method (as described in MPIF Standard 56) to evaluate the endurance limit (σ50, 50% survival) at 2 million cycles and fully reversed loading (R = -1). Further test sample evaluation consisted of Vickers hardness measurements, carbon and oxygen analyses in Leco instruments, sintered density measurements using the Archimedes' principle, and metallographic studies by light optical microscopy.

3. Results

3.1 Density, hardness and chemistry

Results from the analyses performed on the sinter-hardened test specimens are presented in Table 3.

All materials have slightly more than 0.1 g/cm^3 higher sintered density after high temperature sintering (SH-1250) than after sintering at 1120 °C (SH-1120). The Ni-containing materials have highest density values and density increases with higher Ni content. For the Cu-containing materials the density decreases somewhat with a higher Cu content. The different materials have relatively comparable hardness (361-391 HV10) after process SH-1120. Considerably higher hardness values (425-457 HV10) were obtained after process SH-1250, and also here the differences between the materials are small. The sintered carbon contents are close to 0.55% C for the CrA-based materials and close to 0.45% C for the CrM-based materials after both processes. The materials have comparable sintered oxygen contents which are considerably lower after process SH-1250 than after process SH-1120.

Table 3. Sintered density (SD), hardness (HV10) and C/O contents (in wt.%) of sinter-hardened test bars.

Material	Process	SD (g/cm^3)	HV10	C (%)	O (%)
CrA-1Cu	SH-1120	7.06	388	0.54	0.08
	SH-1250	7.17	457	0.57	0.02
CrA-2Cu	SH-1120	7.02	390	0.56	0.09
	SH-1250	7.15	448	0.57	0.02
CrA-1Ni	SH-1120	7.10	361	0.56	0.08
	SH-1250	7.21	439	0.55	0.02
CrA-2Ni	SH-1120	7.13	391	0.56	0.08
	SH-1250	7.25	425	0.57	0.02
CrM	SH-1120	7.00	374	0.45	0.10
	SH-1250	7.12	438	0.48	0.03

3.2 Fatigue strength

The fatigue strength values of the evaluated materials are shown in Figure 1, where the fatigue strength is expressed as the endurance limit (σ50) obtained in the plane bending fatigue testing. The conventional sinter-hardening process (SH-1120) resulted in strength values between 310-335 MPa. With the integrated high temperature sintering and sinter-hardening process (SH-1250), the fatigue strength is 10-15 % higher for all materials with values in the range of 340-365 MPa. The differences in fatigue strength between the different materials after each process are relatively small.

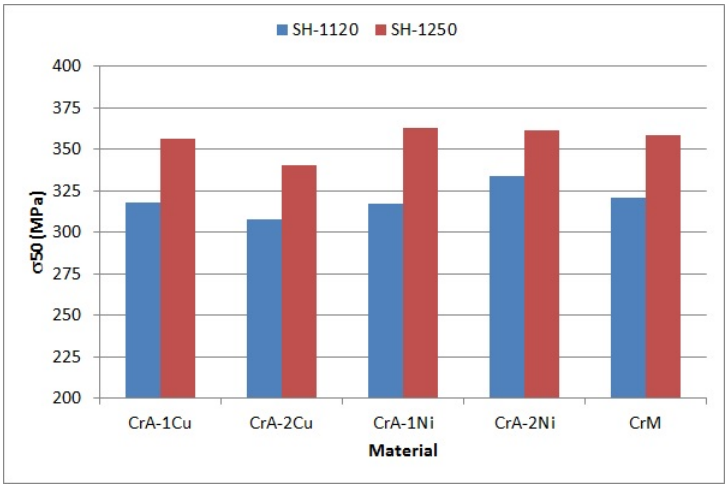


Fig. 1. Fatigue strength of evaluated materials after different sinter-hardening trials.

3.3 Microstructures

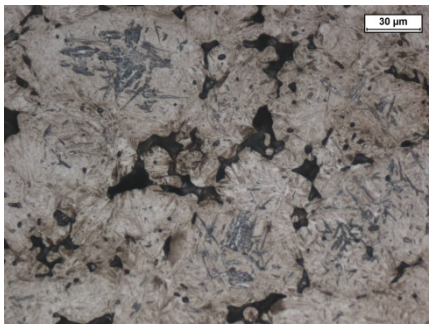
All materials have predominantly martensitic microstructures after both sinter-hardening processes, as shown in Figure 2. After process SH-1120, materials CrA-1Cu and CrA-1Ni contain 15-20 % bainite whereas materials CrA-2Cu and CrA-2Ni contain 5-10 % bainite mixed with the martensite. These materials contain only small amounts (<5 %) of bainite after process SH-1250. The Ni-containing materials also have small areas of Ni-rich austenite in the microstructure after process SH-1120, but not after process SH-1250. The CrM material is fully martensitic after both processes. There are no significant differences in microstructure composition between center and surface regions of the test specimens in the materials that were sinter-hardened in process SH-1120. However, all materials that were sinter-hardened in process SH-1250 have slightly coarser martensite together with some retained austenite at the specimen surfaces. The retained austenite appears primarily in the specimen corners (as illustrated in Figure 3). The different test materials display similar pore structures, with irregular pore shape after process SH-1120 and more rounded pore shape after process SH-1250 (see Figure 2).

4. Discussion

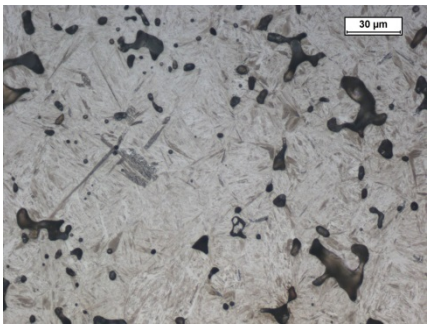
The results from this investigation demonstrate that the evaluated Cr-alloyed powder grades can provide very good fatigue properties after sinter-hardening, which is in line with results from earlier studies [6, 7]. The obtained fatigue strength values are 310-335 MPa after sinter-hardening in a mesh belt sintering furnace and 340-365 MPa after sinter-hardening in a roller hearth high temperature sintering furnace. For all five tested materials, 10-15 % higher fatigue strength was achieved with the combined high temperature sintering and sinter-hardening process compared to the conventional mesh belt sinter-hardening process. The higher sintering temperature in the former process gave test specimens with higher sintered density (~0.1 g/cm^3) and more rounded pore structures. Both are effects that contribute to the enhancement of fatigue properties.

The microstructures of the sinter-hardened test specimens are predominantly martensitic for all materials after both processes. For the Astaloy CrA materials with 1% Cu/Ni additions, the microstructures contain up to 20 % bainite mixed with the martensite after the conventional sinter-hardening. There was less bainite (up to 10 %) in the microstructures of the Astaloy CrA materials with 2% Cu/Ni additions after this process, which shows the hardenability effect of increasing the Cu/Ni content. However, in all Astaloy CrA based test materials the microstructures are nearly fully martensitic (<5 % bainite) after sinter-hardening in the high temperature furnace, although the same cooling rate was applied in both processes and with basically the same sintered carbon content (around 0.55% C) in all materials. This is an effect of a more homogenous distribution of Cu and Ni, respectively, in the materials due to more effective diffusion of the alloying elements at a higher sintering temperature. The microstructures of the Astaloy CrM material are fully martensitic after both sinter-hardening processes, which demonstrate the high hardenability of this grade at relatively low sintered carbon content (around 0.45% C) and without any extra alloying additives.

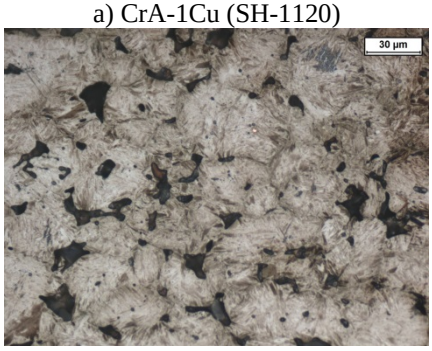
There are relatively small differences in fatigue strength between the different test materials after each respective sinter-hardening process. Hence, the differences in microstructure composition discussed above have no significant effect on the fatigue properties. This shows that the addition of either 1% Cu or 1% Ni to Astaloy CrA is enough to reach high fatigue performance after sinter-hardening with a cooling rate of 5 °C/s and with a sintered carbon content of 0.55% C. Increasing the alloying addition from 1 to 2% Cu shows a tendency to decrease the fatigue strength, which should be due to the swelling effect of Cu during sintering leading to lower sintered density. The opposite tendency with a slight increase in fatigue strength is seen when increasing the alloying addition from 1 to 2% Ni, which can be linked to the densifying effect of Ni during sintering giving higher sintered density. It can be noted that Astaloy CrM with 0.45% C in sintered carbon content provides equivalent fatigue strength to the Astaloy CrA materials with Cu/Ni additions after both sinter-hardening processes, although the Astaloy CrM specimens have lower sintered density.



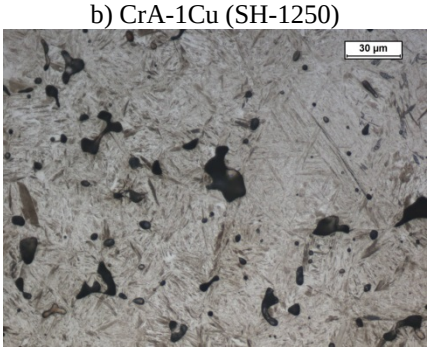
a) CrA-1Cu (SH-1120)



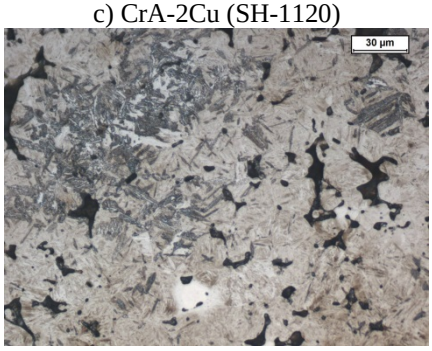
b) CrA-1Cu (SH-1250)



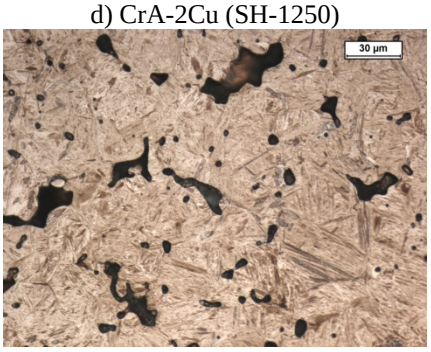
c) CrA-2Cu (SH-1120)



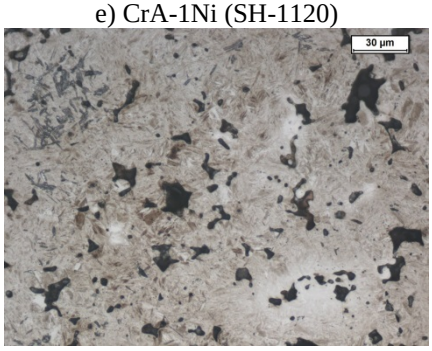
d) CrA-2Cu (SH-1250)



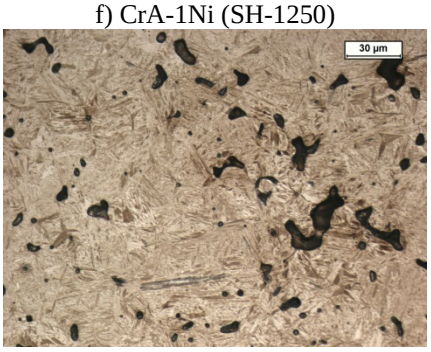
e) CrA-1Ni (SH-1120)



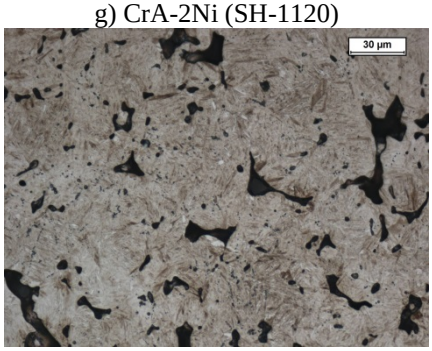
f) CrA-1Ni (SH-1250)



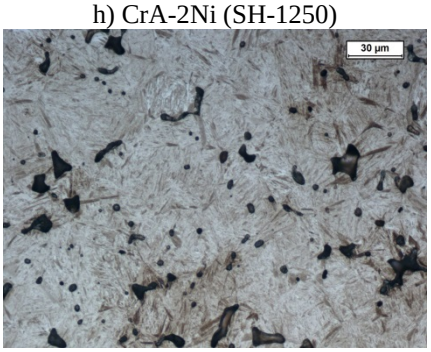
g) CrA-2Ni (SH-1120)



h) CrA-2Ni (SH-1250)

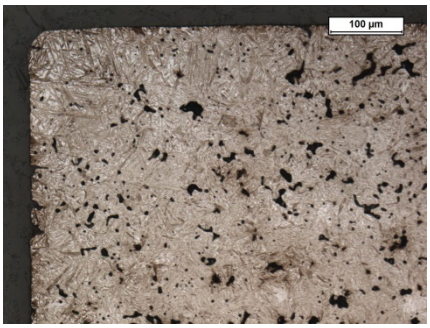


i) CrM (SH-1120)



j) CrM (SH-1250)

Fig. 2. Microstructures in the center of sinter-hardened test bars (etched in 25 % Nital – 75 % Picral).



CrA-2Ni (SH-1250)

Fig. 3. Microstructure in corner of sinter-hardened test bar (etched in 25 % Nital – 75 % Picral).

In the sinter-hardening trials of this investigation, small additions of hydro-carbons are used in the N_2/H_2 sintering atmospheres to counteract decarburization of the test samples. Some decarburization still occurs since carbon acts as a reducing agent on oxides in the materials during sintering. More effective oxide reduction is achieved at higher temperatures leading to more carbon loss, which is why higher graphite contents are used in the test materials for the high temperature sinter-hardening trial. This carbon loss due to oxide reduction leads to an even decarburization throughout the test specimens during sintering. However, having some carbon potential in the sintering atmosphere is crucial for preventing surface decarburization of the test specimens, since this will deteriorate the bending fatigue properties. There are no signs of surface decarburization in the conventionally sinter-hardened test materials, which shows that the CH_4 addition to the atmosphere has given satisfactory carbon control. The use of the minor C_3H_8 addition to the atmosphere in the high temperature sinter-hardening trial has on the other hand resulted in some surface carburization of the test specimens. This is the explanation for the appearance of coarser martensite and some retained austenite at the specimen surfaces, and also the reason for the considerably higher hardness values compared to the conventionally sinter-hardened materials. Slight carburization can be positive for the bending fatigue properties of sinter-hardened PM parts, since compressive stresses are formed at the part surfaces which enhance the fatigue strength [8]. Although, the effect on the fatigue properties is unclear in the present investigation as the retained austenite in the specimens show that there has been some over-carburization during sintering. The results rather suggest that even higher fatigue properties should be attainable with Cr-alloyed PM steel after an integrated high temperature sintering and sinter-hardening process, by adjusting the carbon potential of the sintering atmosphere to optimize the carbon content at the specimen surfaces. There are atmosphere control systems available for achieving optimum carbon control during high temperature sintering [11].

5. Conclusions

- Powder grades pre-alloyed with Cr are suitable for cost-efficient manufacturing by sinter-hardening of PM steel components that require high fatigue strength. Astaloy CrA (1.8% Cr), with addition of 1-2% Cu or Ni and 0.55% C as-sintered, and Astaloy CrM (3% Cr-0.5% Mo), with 0.45% C as-sintered, provide high and comparable fatigue strength after sinter-hardening.
- Bending fatigue strength values of the Cr-alloyed PM steels measured 310-335 MPa after sinter-hardening in a mesh belt furnace with a 1120 °C sintering temperature and a cooling rate of 5 °C/s (at sintered density 7.0-7.1 g/cm^3). 10-15 % higher fatigue strength in the range of 340-365 MPa are obtained after sinter-hardening in a roller hearth high temperature furnace with 1250 °C in sintering temperature and the same cooling rate (at sintered density 7.1-7.2 g/cm^3).

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