EFFECT OF COMPOSITION AND PROCESSING ON THE PRECISION OF SINTER HARDENING POWDER METAL (PM) STEELS

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ABSTRACT

Sinter hardening is commonly used in the powder metal (PM) industry as a cost effective method to produce components requiring high mechanical performance. Using accelerated cooling in combination with higher alloying contents, sinter hardening steels offer similar performance to conventional heat treated steels with reduced distortion. As demands on component precision increase, understanding the effects of processing and composition on the precision of the sinter harden steels will be required by component manufacturers to select the proper sinter hardening grade and composition for a given set of requirements. In this paper, common sinter hardening alloys are subjected to various processing conditions. The mechanical performance and precision of the different compositions under these processing conditions will be presented.

INTRODUCTION

Sinter hardening is defined as a thermal process in which a ferrous material is sintered and cooled at a rate sufficient enough to produce a predominately martensitic microstructure. To achieve the necessary cooling rates, convective cooling units are typically installed in the cooling zone of the sintering furnace to produce the accelerated cooling rates. When used with specially designed alloy systems, martensite transformation can be achieved.

Specially designed prealloyed systems for sinter hardening have traditionally used nickel and molybdenum alloying along with elemental copper additions to provide sufficient hardenability. These alloying elements provide the necessary hardenability while also providing a robust material which can be used in a variety of sintering atmospheres. With the advent of nitrogen / hydrogen based sintering atmospheres, Höganäs introduced chromium based alloying systems to utilize the hardening characteristics of chromium. The sintering of chromium requires good control of the oxygen level in the atmosphere in order to avoid oxidation of the chromium in the alloy. Therefore, endogas atmospheres should be avoided due to their high oxygen potential. The nitrogen / hydrogen atmospheres used in the
PM industry today have sufficiently low oxygen potentials and allow for the successful sintering of chromium containing materials.

Copper is commonly used in conjunction with iron or alloy steel powders to increase hardenability. When sintered, liquid copper penetrates the iron grain boundaries and swelling occurs. Many studies have shown that when carbon and copper are both present, an interaction between the two elements takes place during sintering. Carbon, which has a high solubility in iron, decreases swelling due to grain boundary penetration by molten copper. Increasing the carbon level, therefore, can be used to reduce the amount of dimensional change.

Since traditional sinter hardening alloying systems rely on copper additions and increased carbon contents, segregation of these admixed elements can lead to variation in the final product. Chromium materials, depending on their alloying, do not require high graphite levels or copper additions to achieve high hardenability. This makes them attractive options with regards to dimensional stability. Dimensional stability of sinter hardening alloys is critical since adjustments for dimensions using secondary machining processes will require either advanced machining enhancers or low productivity machining processes.

In this paper, the dimensional stability of common industry sinter hardenable grades will be examined to understand the performance and sensitivity of the different grades to composition and processing. While dimensional change can be influenced by a number of factors including density, sintering time and temperature, in this study only the material composition and cooling rate were varied. The dimensional change, dimensional stability, microstructure and mechanical performance of the alloys will be presented.

**EXPERIMENTAL PROCEDURE**

Three commercially available base irons were evaluated in this study. The compositions of the base irons evaluated are shown in Table 1.

<table>
<thead>
<tr>
<th>Base Iron</th>
<th>MPIF Designation</th>
<th>Ni</th>
<th>Mo</th>
<th>Mn</th>
<th>Cr</th>
</tr>
</thead>
<tbody>
<tr>
<td>Astaloy® A</td>
<td>FL-4600</td>
<td>1.9</td>
<td>0.55</td>
<td>0.2</td>
<td>-</td>
</tr>
<tr>
<td>Astaloy® MoNi</td>
<td>FL-4800</td>
<td>1.4</td>
<td>1.2</td>
<td>0.4</td>
<td>-</td>
</tr>
<tr>
<td>Astaloy CrM®</td>
<td>FL-5300</td>
<td>-</td>
<td>0.5</td>
<td>0.1</td>
<td>3.0</td>
</tr>
</tbody>
</table>

The alloying technique used for all three base irons is prealloying where the alloying elements are added during the melt before atomization. While this evenly distributes the alloying elements over the entire powder particle, it also increases the hardness of the powder particle which reduces compressibility compared to a pure iron particle. The compressibility curve for each alloy is shown in Figure 1. Die wall lubrication was used for the generation of the compressibility curves. A compressibility curve for a compressible base iron (ASC100.29) is shown to illustrate the decrease in compressibility due to the alloying method.
To the base irons listed in Table 1, premixes were manufactured in the North American Höganäs Pilot Mixing Center. To the FL-4600 & FL-4800 base irons, three levels of copper (0%, 1% 2%) and three levels of graphite (0.3%, 0.6%, 0.9%) were added for a total of 18 mixes. Since the FL-5300 base iron does not require a copper addition, only the three levels of graphite were added for a total of three mixes. For all of the premixes, the same elemental copper (Cu-165 ACu Powder), natural graphite (Asbury 1651), and lubricant (Intralube® E, Höganäs AB) were used.

Standard transverse rupture strength (TRS) specimens were compacted for the mixes which contained only graphite to a green density of 6.95 g/cm$^3$. The copper containing mixes were compacted to a green density of 7.0 g/cm$^3$. This was done in order to obtain a sintered density of 6.9 g/cm$^3$ for all of the materials. The specimens were sintered in a mesh belt furnace for 30 minutes at 1120 °C (2050 °F) in a 90% nitrogen / 10% hydrogen atmosphere. The TRS specimens were cooled at two different cooling rates which were measured between 704 °C (1300 °F) and 315 °C (600 °F): 0.7 °C/s (1.4 °F/s) and 2 °C/s (4 °F/s). The specimens which were subjected to the accelerated rate were subsequently tempered for 1 hour at 204 °C (400 °F) in air.

After processing, the specimens were measured for dimensional change (die to sinter), sintered density, sintered carbon, apparent hardness, and transverse rupture strength according to industry standards. Metallographic analysis was also performed on selected specimens to understand the microstructural changes that occurred during processing.

**RESULTS**

The effect of carbon and copper content in the as sintered condition on the three base irons is shown in Figure 2.

![Figure 2. Dimensional change of alloys with various carbon and copper contents at 0.7 °C/s (1.4 °F/s) cooling rate](image-url)
All of the alloy systems increase in growth as the carbon content increases. Both the FL-4600 & FL-4800 alloys increase linearly over the carbon range. The FL-5300 increases linearly between 0.2% and 0.6% carbon. At the higher graphite levels, the amount of growth does not change substantially from the 0.6% addition. For the FL-4600 and FL-4800, the interaction between the carbon and copper can be observed. For both the FL-4600 and FL-4800 with 1% copper, the dimensional change is not as sensitive to carbon. For the FL-4800 with 2% copper addition, increasing carbon content decreases the amount of growth observed. The FL-4600 with 2% copper shows a more dramatic drop in growth between 0.6% and 0.8% carbon compared to the FL-4800 alloy over the same carbon range.

Transverse rupture strength results for the alloys are shown in Figure 3.

![Figure 3](image-url)

**Figure 3.** Transverse rupture strength of alloys with various carbon and copper contents at 0.7 °C/s (1.4 °F/s) cooling rate

Different responses to copper and graphite were observed for the alloy systems. The FL-4600 showed a linear increase in strength with increasing carbon level. When copper was added, a large increase in strength was observed. For both copper levels, the highest strength level was observed with a carbon content of 0.5%. As the carbon level increased from 0.5%, a significant drop in strength was observed. The FL-4800 alloy had a slightly different response. Only a large increase in strength was observed for the 1% copper addition. The same trend with decreasing strength as carbon level increased was observed. The 2% copper addition and no copper addition showed similar strength levels. Similar strength levels were observed for these two materials over the carbon range. The FL-5300 material showed a substantial decrease in strength at the higher carbon levels. Increasing the carbon from 0.5% to 0.8% decreased the strength from 1600 MPa to 800 MPa.

Microstructure analysis provides insight into the dimensional and strength responses of the materials in the as sintered condition. Microstructure photographs for the FL-4600 material are shown in Figure 4. With no copper addition, the FL-4600 maintains a bainitic microstructure. As the carbon increases, the bainite becomes denser. The copper additions to this alloy promote the formation of martensite. Even at the lowest carbon level, both the 1% and 2% copper additions result in martensite formation. Increasing graphite increases the amount of martensite formation. Comparing the 1% and 2% copper additions and correlating to the strength results, the large drop in strength from when the graphite level increases from 0.5% to 0.8% is due to the amount of martensite and retained austenite formed. The retained austenite was observed between the martensite plates and especially where the copper concentration is high – at the grain boundaries.
Figure 4. As sintered photomicrographs of FL-4600 with various carbon and copper contents at 0.7 °C/s (1.4 °F/s) cooling rate

The photomicrographs for the FL-4800 material are shown in Figure 5. The alloying of this material system is higher than the FL-4600 and hence martensite transformation occurs at lower alloying levels. Martensite was observed at a 0.8% carbon level. Copper additions allow for the martensite to form at lower carbon levels. With 1% copper added, the amount of martensite in the structure was 50% at the 0.5% carbon level. Increasing the copper to 2%, the level of martensite at the 0.5% carbon level was 95%. Retained austenite was observed for the FL-4800 material especially at the higher carbon levels with the copper addition. With a 1% copper addition, a large increase in retained austenite was observed as the carbon increased from 0.5% to 0.8%. This resulted in a significant decrease in strength. At the 2% copper level, the FL-4800 maintains a similar strength level across the carbon range. It should be noted that this strength decrease is not unexpected for either Ni-Mo prealloyed system. With copper additions and high carbon contents, these alloys should be tempered to maximize strength even in the as sintered condition.
The FL-5300 material at the lower carbon content is bainitic material. Increasing the carbon transforms the microstructure to martensite. However, since the carbon level is lower, the amount of retained austenite formed is negligible. At the higher carbon level, the FL-5300 material is martensitic with a high...
amount of retained austenite which results in the dramatic reduction in strength observed. Since the
hardenability of this alloy is high, carbon levels above 0.6% are not recommended to maximize
mechanical performance.

Since these materials are primarily used for sinter hardening applications, it is interesting to understand
the effect of increased cooling rate on the dimensional and mechanical performance. The dimensional
change at 2.0 °C/s (4.0 °F/s) for the different alloys is shown in Figure 7. The black lines represent the
tempered measurements.

Tempering of martensitic steels is a common practice to improve the physical properties as well as the
dimensional stability. Tempering involves raising the temperature of steel to a value below the eutectoid
temperature for a fixed amount of time and then cooling to room temperature. The intent is to allow
diffusion processes time for the martensite to change from its body center tetragonal (BCT) structure to
ferrite (BCC) plus carbide. This change increases the density which in turn reduces the dimensional
change.

In Figure 7, this process is illustrated. When the materials become martensitic, the tempering operation
reduces the dimensional change compared to the sinter hardened condition. For the FL-4600 with no
copper, tempering had no effect on the dimensional change. With the copper additions and higher carbon
levels, tempering reduces the dimensional change of the FL-4600 material. The FL-4800 material without
copper shows a reduction in dimensions only at the higher carbon level. A large reduction is observed
when the copper addition is made to the FL-4800 alloy. The FL-5300 material showed a similar trend
with tempering decreasing dimensions at the middle and higher carbon level.

The microstructure for each material in the sinter hardened condition correlates to the dimensional change
reduction observed after the tempering operation. The microstructures for the FL-4600 in the sinter
hardened condition are shown in Figure 8.
A reduction in dimensional change was only observed with the copper additions and the higher carbon levels for the FL-4600. Under these conditions, the FL-4600 is predominately martensitic material. The hardenability of the alloy is not high enough to form martensite with only carbon. Since no martensite was formed, tempering has no effect on the dimensional change.

The microstructures for the FL-4800 material in the sinter hardened condition are shown in Figure 9. This alloy has a higher hardenability than the FL-4600, so martensite is formed without a copper addition. At the 0.5% carbon level, martensite is formed. In the dimensional analysis, a reduction in dimensional change after tempering was observed above 0.5% carbon. With copper additions, martensite is easily formed in the FL-4800. Even at 0.25% carbon, some martensite is formed and tempering slightly reduces the growth. As the carbon level increases, the microstructure becomes entirely martensitic and a larger reduction in growth is observed after tempering.
The microstructures of the FL-5300 material are shown in Figure 10.

Only at the higher carbon levels does tempering reduce the growth of the FL-5300 where martensite is generated. The bainitic structure at 0.25% carbon was not affected by the tempering operation. Tempering also improves the mechanical performance as well as reduces growth. The effect of tempering on the transverse rupture strength of the alloys is shown in Figure 11.
For the FL-4600 with no copper addition, the tempering did not have an appreciable effect on the strength since no martensite was formed. However, for the martensitic structures, a large improvement in strength was observed. For the higher carbon levels, strength levels increased 600 – 900 MPa (87,000 – 130,000 psi). The effect of tempering on the FL-4800 alloy was especially dramatic on the 2% copper addition material. The strength increased 800 MPa (116,000 psi) at the higher graphite levels illustrating the need to temper this alloy after sinter hardening. The FL-5300 alloy improved significantly at the higher carbon level. At the 0.5% carbon level a slight increase was observed even though this was an entirely martensitic microstructure.

Based on the initial study, the alloys optimum composition in the sinter hardened condition was chosen to understand the dimensional stability of the alloys over a number of components and sintering trials. The materials chosen are listed in Table 2.

Table 2. Compositions selected for dimensional stability study

<table>
<thead>
<tr>
<th>Base material</th>
<th>Copper</th>
<th>Carbon</th>
</tr>
</thead>
<tbody>
<tr>
<td>FL-4600</td>
<td>2</td>
<td>0.8</td>
</tr>
<tr>
<td>FL-4800</td>
<td>2</td>
<td>0.8</td>
</tr>
<tr>
<td>FL-5300</td>
<td>-</td>
<td>0.5</td>
</tr>
</tbody>
</table>

It should be noted that while similar strength results may have been obtained for the FL-4600 & FL-4800 material at lower copper levels, the higher copper level was chosen due to the higher hardenability. In industry, this copper level is chosen in order to obtain a completely martensitic microstructure in larger parts where mass effect has to be considered. For the FL-5300, no benefit to strength was observed at the higher carbon level.

For each mix, 55 mm OD x 35 mm ID x 20 mm height (2.16 in OD x 1.38 in ID x 0.79 in. height) rings were compacted to achieve a sintered density of 6.9 g/cm³. Rings were randomized and sintered in a mesh belt furnace for 30 minutes at 1120 °C (2050 °F) in 90% nitrogen / 10% hydrogen atmosphere. An accelerated cooling rate of 2 °C/s (4 °F/s) was used to achieve a martensitic microstructure. Each sintering run contained 30 rings for each material.

Each ring was marked with an “x” parallel to the filling direction for orientation. The outer diameter of the rings was measured parallel to the filling direction and perpendicular to the filling direction. Out of roundness was calculated according the Equation 1.

$$OOR = \text{Absolute Value} (OD_{parallel} - OD_{perpendicular}) \quad (Eq.1)$$
The rings were measured after tempering which was conducted at 204 °C (400 °F) for one hour in air. Multiple sintering runs were conducted using the above procedure.

The results of the dimensional stability analysis are shown in Figure 12.

**Figure 12.** Out of round measurements

The two alloys which utilized a copper addition had similar scatter in the out of round measurements. The FL-5305 alloy which does not require a copper addition to sinter hardened had a drastically reduced out of round scatter. Similar results were achieved for all alloys across the sintering runs.

According to the results achieved in the TRS bar study on dimensions, the FL-5305 and FLC2-4808 alloy should have a similar dimensional change after sinter hardening and tempering: approximately 0.10%. By eliminating the copper addition, an increase in precision was achieved for the FL-5305 alloy. This was done while achieving the same strength properties as the FLC2-4808.

**CONCLUSIONS**

The following conclusions were drawn from this study:

- The properties of sinter hardening alloys can be manipulated through chemistry (carbon & copper) and processing. Depending on the mass of the part and hardenability required, optimization of the alloys can be made to provide increased strength or lower dimensional growth.

- The amount of martensite generated during the sinter hardening process will play a role in the final dimensional change of the component. As the martensite content of the alloy increases, the amount of growth increases. To control the amount of growth in sinter hardening alloys which require a copper addition, increasing the graphite will decrease the amount of growth due to the interactions between the copper and carbon. Tempering of the high carbon alloys resulted in a reduction of dimensional growth.
change and an increase in strength for all of the alloys and combinations where retained austenite was
generated. Tempering increased the strength of the alloys 600 – 900 MPa depending on the copper
and carbon content.

- The FL-5305 alloy had the highest precision of the alloys investigated with regards to out of round
measurements. The increase in precision was achieved without sacrificing strength. The optimal
carbon level for this alloy to achieve good precision and strength was determined to be 0.5%. At this
carbon level, the FL-5305 had 1/3 of the dimensional variation compared to the FLC-4608 and FLC2-
4808.

FUTURE WORK

This study investigated the general interactions between copper and carbon with common sinter
hardening grades. Future work will examine this interaction in more depth looking at the response of
different graphite and copper powders on the precision of these alloys. Methods to improve the stability
of the alloys will also be investigated.

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REFERENCES

ASTM International, West Conshohocken, PA.

2. R.L. Lawcock, “Effect of carbon on dimensional and microstructural characteristics of Fe-Cu


Bonded Mixes”, Advances in Powder Metallurgy & Particulate Materials, compiled by C. Ruas and T.A.

5. B. Hu, R. Warzel III, et al, “Development of A New Machinability Enhancing Additive for Sinter-
hardened and Heat-treated PM materials”, Advances in Powder Metallurgy & Particulate Materials,
compiled by R. Lawcock, A. Lawley and P. McGeehan, Metal Powder Industries Federation, Princeton,

