Effect of Heat Treatment and Case Carburizing High-Density P/M Steels

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ABSTRACT

Recent advancements in high-density lubricants enable P/M steels to be processed to densities approaching 7.40 g/cm³ in a single compaction step using heated compaction tools via the AncorMax D® process. This broadens the number of suitable applications for P/M steels, including high performance gears. However, in addition to high-density, the microstructural and mechanical property requirements vary depending on the application. This objective of this paper is to quantify and understand the mechanical property differences obtained by subjecting high-density P/M steels to various heat-treatments. Mechanical properties, fatigue properties, and microstructural analysis will be presented in the as sintered, quenched and tempered, and carburized states.

INTRODUCTION

The introduction of high density compaction technology, known as the AncorMax D process enables P/M part producers to achieve up to 98% pore-free densities in green parts [1]. This premix technology features a proprietary lubricant/binder system requires a high compaction pressure of 550 – 823 MPa (40 – 60 tons/in²). Die temperatures must be maintained in the range 60 – 70 °C (140 – 150 °F) and this is achieved using cartridge heaters incorporated within the die. Warm die compaction, coupled with advanced lubricant/binder systems has been shown to increase green densities without the need to heat the powder [2].

Carburizing is the addition of carbon to the surface of low-carbon steels at temperatures generally between 850 and 950 °C (1560 and 1740 °F), at which austenite, with its high solubility for carbon, is the stable crystal structure. Hardening is accomplished when the high-carbon surface layer is quenched to form martensite so that a high-carbon martensitic case with good wear and fatigue resistance is superimposed on a tough, low-carbon steel core. [3]

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Case depth of carburized steel is a function of carburizing time and the available carbon potential at the surface. [4] When prolonged carburizing times are used for deep case depths, a high carbon potential produces a high surface-carbon content, which may result in excessive retained austenite or free carbides. These two microstructural elements both have adverse effects on the distribution of residual stress in the case-hardened part. Consequently, a high carbon potential may be suitable for short carburizing times but not for prolonged carburizing. The purpose of this paper will be to investigate the performance levels achievable of FLN2 – 4405 by using conventional sintering, and secondary heat-treatments such as quench and tempered, and case carburizing.

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EXPERIMENTAL PROCEDURE

The press-ready, binder-treated material was tested in a production environment to evaluate its properties through a single press/single sinter, quench and tempered, and the case carburized condition. The base powder Ancorsteel 85HP is water atomized and pre-alloyed with 0.85 w/o molybdenum. The base iron is premixed with 2.0 w/o nickel and 0.40 w/o graphite. The nickel used was INCO 123 and the graphite was Asbury 3203H. The compositions of the test materials that were evaluated in this study are listed in Table I.

<table>
<thead>
<tr>
<th>Base Powder</th>
<th>Mo</th>
<th>Nickel</th>
<th>Graphite</th>
<th>lube organics w/o</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ancorsteel 85HP</td>
<td>0.85</td>
<td>2.0</td>
<td>0.40</td>
<td>0.55</td>
</tr>
</tbody>
</table>

TEST SPECIMEN / COMPACTION AND SINTERING

Transverse rupture, tensile dog-bone, impact samples were compacted at pressures varying from 415 to 760 MPa, (30 to 60 tsi) (Fatigue samples were compacted to a density of 7.20 g/cm³) compaction dies were heated to 63 °C (145 °F).

Green density, sintered density, and transverse rupture strength was determined from the average of five compacted transverse rupture (TRS) specimens (ASTM B-528). Tensile strength, yield strength, and maximum elongation were obtained from the average of five dog-bone tensile samples (ASTM E-8). Impact energy was determined from the average of five un-notched Charpy Impact bars (ASTM E-23). Apparent hardness measurements were performed on the surface of the TRS bars using a Rockwell hardness tester. All measurements were conducted using the HRA scale for ease of comparison.

All test pieces were sintered under production conditions in an Abbott continuous belt high temperature furnace at the Hoeganaes R&D facility, in Cinnaminson, NJ. The sintering condition used for the test specimen is listed below.

Sintering Temperature: 1120 °C (2050 °F)
Atmosphere: 90 v/o N₂ - 10 v/o H₂
Time in Hot Zone: 20 minutes

For the samples that were carburized, the parameters are listed below.

Temperature: 925 °C (1700 °F)
Time at Temperature: 240 minutes
Quench: Pressure / Nitrogen
Tempering: 205 °C (400 °F) for 1 hour

For the samples that were quenched and tempered, the parameters are listed below.

Temperature: 900 °C (1650 °F)
Atmosphere: 75 v/o H₂ – 25 v/o N₂
Time at Temperature: 50 minutes
Oil Quench: 75 °C (165 °F)
Tempering: 205 °C (400 °F) for 1 hour
Tensile testing was performed on a 267,000 N (60,000 lb.) Tinius Olsen universal testing machine with a cross-head speed of 0.635 mm/min (0.025 in/min). Elongation values were determined by utilizing an extensometer with a range of 0 - 20%. The extensometer was attached to the samples up to failure.

Rotating bending fatigue samples were pressed to a density of 7.20 g/cm$^3$, and machined from blanks. The as-sintered fatigue samples were machined and ground to size after the sintering operation. The heat-treated fatigue samples were rough machined following sintering then heat treated, finished ground, and polished to size. The dimensions of the specimen used for this analysis, along with allowable dimensional tolerances, are shown in Figure 1.

Fatigue testing was performed on six randomly selected Fatigue Dynamics RBF-200 machines at a rotational speed of 8000 rpm. These rotating bending machines are of the mechanical and non-resonant type and are an efficient means of inducing fatigue in a specimen of round cross section. [5] A staircase method was used utilizing 30 samples and a run-out limit of $10^7$ cycles. The staircase method of testing was regulated so that there were both failures and run-outs at a minimum of two stress levels. [6] The percentage of failures for each stress level was calculated and plotted on a log-normal graph. From these plots, the fatigue endurance limit (FEL) at 50% and 90% was determined by linear extrapolation. The 50% FEL represents the stress level where 50% of the specimens will break and 50% will run-out. The 90% FEL represents the stress level where 90% of the specimens will "run-out" and 10% will break.

![Figure 1 Dimensions of Rotating Bending Fatigue Specimen](image)

**RESULTS AND DISCUSSION**

Tensile strength for the “as-sintered”, carburized, and quench & tempered conditions are summarized in Tables II through IV. These tables also include information of green and sintered density, apparent hardness, and impact energy. Fatigue properties are listed in Table V.

In order to establish a frame of reference for the lubricant/binder processing technology, the compressibility of FLN2 – 4405 was compared to a conventionally pressed premix using Acrawax C as the lubricant pressed at room temperature. A compressibility plot for this material is shown in figure 2. With the AncorMax D process, higher green densities were achieved. Green densities ranged from 6.96 – 7.45 g/cm$^3$ for the alloy examined. With the advanced lubricant/binder system compared to a standard compaction process a difference of 0.14 g/cm$^3$ was observed at 415 MPa.
With increasing compaction pressures the difference increased to 0.22 g/cm$^3$ at a pressure of 825 MPa (60 tsi). This indicates that the AncorMax D process is a more effective process for densification at higher compaction pressures.

### Table II. As Sintered Properties

<table>
<thead>
<tr>
<th>Compaction Pressure (MPa/ksi)</th>
<th>Green Density (g/cm$^3$)</th>
<th>Sintered Density (g/cm$^3$)</th>
<th>Apparent Hardness HRA</th>
<th>TRS (MPa/10$^3$ psi)</th>
<th>UTS (MPa/10$^3$ psi)</th>
<th>0.20% Offset (MPa/10$^3$ psi)</th>
<th>Elong %</th>
<th>Impact Energy (J/ft*lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>415/30</td>
<td>6.96</td>
<td>6.98</td>
<td>46</td>
<td>1028/147</td>
<td>501/72</td>
<td>357/51</td>
<td>1.8</td>
<td>10/7</td>
</tr>
<tr>
<td>550/40</td>
<td>7.21</td>
<td>7.22</td>
<td>48</td>
<td>1308/187</td>
<td>613/88</td>
<td>412/59</td>
<td>2.7</td>
<td>17/13</td>
</tr>
<tr>
<td>690/50</td>
<td>7.38</td>
<td>7.39</td>
<td>50</td>
<td>1497/214</td>
<td>698/100</td>
<td>471/68</td>
<td>3.2</td>
<td>26/20</td>
</tr>
<tr>
<td>825/60</td>
<td>7.45</td>
<td>7.48</td>
<td>50</td>
<td>1610/231</td>
<td>718/103</td>
<td>503/72</td>
<td>2.3</td>
<td>39/29</td>
</tr>
</tbody>
</table>

### Table III. Carburized Properties

<table>
<thead>
<tr>
<th>Compaction Pressure (MPa/ksi)</th>
<th>Green Density (g/cm$^3$)</th>
<th>Sintered Density (g/cm$^3$)</th>
<th>Apparent Hardness HRA</th>
<th>TRS (MPa/10$^3$ psi)</th>
<th>UTS (MPa/10$^3$ psi)</th>
<th>0.20% Offset (MPa/10$^3$ psi)</th>
<th>Elong %</th>
<th>Impact Energy (J/ft*lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>415/30</td>
<td>6.96</td>
<td>6.99</td>
<td>70</td>
<td>1165/165</td>
<td>766/110</td>
<td>723/104</td>
<td>0.8</td>
<td>8/6</td>
</tr>
<tr>
<td>550/40</td>
<td>7.21</td>
<td>7.25</td>
<td>74</td>
<td>1539/220</td>
<td>984/141</td>
<td>887/127</td>
<td>1.0</td>
<td>13/10</td>
</tr>
<tr>
<td>690/50</td>
<td>7.38</td>
<td>7.39</td>
<td>76</td>
<td>1677/240</td>
<td>1067/153</td>
<td>887/127</td>
<td>0.9</td>
<td>16/12</td>
</tr>
<tr>
<td>825/60</td>
<td>7.45</td>
<td>7.48</td>
<td>77</td>
<td>1838/263</td>
<td>1062/152</td>
<td>914/131</td>
<td>0.9</td>
<td>17/13</td>
</tr>
</tbody>
</table>

### Table IV. Quenched and Tempered Properties

<table>
<thead>
<tr>
<th>Compaction Pressure (MPa/ksi)</th>
<th>Green Density (g/cm$^3$)</th>
<th>Sintered Density (g/cm$^3$)</th>
<th>Apparent Hardness HRA</th>
<th>TRS (MPa/10$^3$ psi)</th>
<th>UTS (MPa/10$^3$ psi)</th>
<th>0.20% Offset (MPa/10$^3$ psi)</th>
<th>Elong %</th>
<th>Impact Energy (J/ft*lbf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>415/30</td>
<td>6.93</td>
<td>6.94</td>
<td>60</td>
<td>1417/203</td>
<td>840/120</td>
<td>806/116</td>
<td>0.9</td>
<td>9/7</td>
</tr>
<tr>
<td>550/40</td>
<td>7.19</td>
<td>7.21</td>
<td>66</td>
<td>1871/268</td>
<td>1059/152</td>
<td>904/130</td>
<td>1.4</td>
<td>15/11</td>
</tr>
<tr>
<td>690/50</td>
<td>7.36</td>
<td>7.35</td>
<td>68</td>
<td>2275/326</td>
<td>1201/172</td>
<td>1052/151</td>
<td>1.3</td>
<td>17/13</td>
</tr>
<tr>
<td>825/60</td>
<td>7.42</td>
<td>7.44</td>
<td>69</td>
<td>2450/351</td>
<td>1273/182</td>
<td>1075/154</td>
<td>1.6</td>
<td>22/16</td>
</tr>
</tbody>
</table>

Sintered densities ranged from 6.98 – 7.48 g/cm$^3$. Figure 3 illustrates the sintered density for both compaction processes as a function of compaction pressure. Increases in sintered densities for AncorMax D were 0.12 g/cm$^3$ at 415 MPa. (30 tsi). With increasing compaction pressures the difference increased to 0.18 g/cm$^3$ at a pressure of 825 MPa (60 tsi) when compared with samples that were conventionally processed.

Shown in Figure 4 and 5 are the effects of compaction pressure on ultimate tensile and yield strengths for the alloy in the as sintered, carburized and heat treated condition. The material developed excellent tensile strength in the quenched and tempered condition. With regards to ultimate tensile strength, increases range from 35 % at 415 MPa. (30 tsi) to 50 % at 825 MPa. (60 tsi) For yield strength, increases > 60 % at 415 MPa. (30 tsi) to > 75 % at 825 MPa. (60 tsi)

The tensile properties of the carburized samples resulted in similar trends; increases in tensile strength were less and range from 13 % at 415 MPa. (30 tsi) to 12 % at 825 MPa. (60 tsi) For yield strength, increases > 50 % at 415 MPa. (30 tsi) to > 45 % at 825 MPa. (60 tsi)
Figure 2: Effect of alloy and processing condition on green density

Figure 3 Effect of processing condition on sintered density (sintered at 1120 °C (2050 °F) - atmosphere of 90 v/o N₂ – 10 v/o H₂) tempered in air at 205 °C (400 °F) for 1 hour
**Figure 4** Effect of processing condition on ultimate tensile strength (sintered at 1120 °C (2050 °F) - atmosphere of 90 v/o N₂ – 10 v/o H₂) tempered in air at 205 °C (400 °F) for 1 hour.

**Figure 5** Effect of processing condition on yield strength (sintered at 1120 °C (2050 °F) - atmosphere of 90 v/o N₂ – 10 v/o H₂) tempered in air at 205 °C (400 °F) for 1 hour.
Rotating bending fatigue data were collected on samples compacted to a density of 7.20 g/cm³. The fatigue endurance limits determined for the material, along with sintered densities are shown in Table V. Figure 6, illustrates the fatigue performance of the alloy tested.

**Table V.** Rotating Bending Fatigue Results – sintered at 1120 °C (2050 °F) - atmosphere of 90 v/o N₂ – 10 v/o H₂

<table>
<thead>
<tr>
<th>Condition</th>
<th>Density (g/cm³)</th>
<th>Survival Limit (MPa/10³psi)</th>
<th>Fatigue Ratio (90% Survival)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As Sintered</td>
<td>7.22</td>
<td>235/34</td>
<td>227/33</td>
</tr>
<tr>
<td>Carburized</td>
<td>7.22</td>
<td>407/59</td>
<td>339/57</td>
</tr>
<tr>
<td>Q/T</td>
<td>7.20</td>
<td>242/35</td>
<td>227/33</td>
</tr>
</tbody>
</table>

**Figure 6.** The relationship of fatigue endurance limits and tensile strength of FLN2-4405

The etched structure of the “as-sintered” sample, which is shown in Figure 7, consist mostly of low carbon divorced pearlite with small regions of bainite. These samples resulted in an apparent hardness at the core of 50 HRA (81 HRB). All core hardness values are listed in table 6. The tensile strengths were minimal, between 501 – 718 MPa (72 –103 ksi). However the low hardness and tensile properties resulted in an increase in elongation and impact properties. Fatigue strength for this condition resulted in 227 MPa (33 ksi) for 90 % FEL (.37 UTS).

The etched structure for the carburized case consist of acicular martensite, nickel rich regions, and areas of retained austenite, shown in Figure 8a & b. The carburized sample resulted in a case depth of about 600 µm from the surface. The core of the carburized sample contains lath martensite, unresolved pearlite, and nickel rich regions, shown in Figure 9. The increase in case carbon from the heat treating resulted in a carbon content of 1.05. The carburized surface increases the case residual compressive stress level and thus improves fatigue endurance limit resistance as well as increasing the tensile properties. These samples resulted in a core hardness of 61 HRA (21 HRC).
Tensile strengths increased up to 1062 MPa (152 ksi). Fatigue strength for this condition resulted in 339 MPa (57 ksi) for 90 % FEL (.34 UTS) a 49% increase over the “as-sintered” samples.

Figure 7. Microstructure of sample “as-sintered” compacted to a density of 7.2 g/cm$^3$, sintered at 1120 °C (2050 °F) - atmosphere of 90 v/o N$_2$ – 10 v/o H$_2$

Figure 8a. b. Microstructure of sample carburized compacted to a density of 7.2 g/cm$^3$, sintered at 1120 °C (2050 °F) - atmosphere of 90 v/o N$_2$ – 10 v/o H$_2$

The etched structure of the quenched & tempered sample is mostly martensitic with nickel rich regions. The martensitic structure is due to the hardenability of the material. This structure is very brittle and resulted in a core hardness of 65 HRA (29 HRC). The tensile strengths were accordingly high; up to 1273 MPa (182 ksi). However the high tensile strength and the highly martensitic structure did not result in high fatigue strength. Fatigue strength for this condition resulted in 227 MPa (33 ksi) for 90 % FEL (.21 UTS).
Figure 9. Microstructure of the core for the carburized sample compacted to a density of 7.2 g/cm³, sintered at 1120 °C (2050 °F) - atmosphere of 90 v/o N₂ – 10 v/o H₂

Table VI. Apparent Hardness Values for Core Samples

<table>
<thead>
<tr>
<th>Core Hardness</th>
<th>As-Sintered</th>
<th>Carburized</th>
<th>Quench &amp; Tempered</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>49 HRA</td>
<td>61 HRA</td>
<td>65 HRA</td>
</tr>
<tr>
<td></td>
<td>79 HRB</td>
<td>21 HRC</td>
<td>29 HRC</td>
</tr>
</tbody>
</table>

Figure 9. Microstructure of the core for the quench and tempered sample compacted to a density of 7.2 g/cm³, sintered at 1120 °C (2050 °F) - atmosphere of 90 v/o N₂ – 10 v/o H₂
CONCLUSIONS

This objective of this paper was to quantify and understand the mechanical property differences obtained by subjecting high-density P/M steels via the AncorMax D® process to various heat-treatments.

- The AncorMax D process via warm die techniques illustrated its ability to increase green and sintered densities thus resulting in higher mechanical properties when compared to conventional pressed material. Increases in green densities ranged from 0.14 – 0.22 (g/cm³), and 0.12 – 0.18 (g/cm³) for the sintered densities were observed.

- At a nominal density of 7.2 g/cm³, FNL-4405 exhibit the highest fatigue limit for the carburized condition. The carburization process resulted in a microstructure of martensite, retained austenite, and nickel rich regions within a 600 micron case. The core of the sample consist of martensite, unresolved pearlite, and nickel rich regions. The carburized surface increases the case residual compressive stress level and thus improves fatigue endurance limit resistance as well as increasing the tensile properties. This resulted in an increase of 45% in tensile strength to 1062 MPa (152 ksi), 80% increase in yield to 914 MPa (131 ksi), and a 49 % increase in fatigue endurance limits to 407 MPa (59 ksi) (.41 UTS) when compared to as-sintered condition.

- The quench and tempered samples resulted in a highly martensistic structure, increases on tensile strengths up to 75% to 1273 MPa (182 ksi) Yield strengths more than doubled to 1075 MPa (154 ksi). Fatigue strength for this condition resulted in 227 MPa (33 ksi) for 90 % FEL.

ACKNOWLEDGEMENTS

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REFERENCES