Sintering Furnace Cycle Influence on Sinter Hardened Part Parameters

PM³TEC 2001
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ABSTRACT:

It is well known that the cooling rate influences the final microstructure and therefore the properties of sinter hardened steel parts. This investigation looked at the relationship between the sintering furnace cycle (time, temperature and atmosphere) and cooling rate to part microstructure. The objective was to identify how the above sintering variables influenced the development of martensite, hardness and strength.

Tests were conducted varying the sintering cycle. The changes to the cycle were sintering time, sintering temperature, atmosphere composition (specifically hydrogen concentration), and cooling rate. The final microstructure was evaluated and correlated to the sintering cycle.

INTRODUCTION:

Sinter hardening has become a very attractive processing technique due to the resulting beneficial mechanical properties and production advantages due to the elimination of some secondary operations. Much knowledge about sinter hardening has been gained as more companies use this process to sinter production parts. Cooling rate is one known factor that influences the as sintered microstructure and ultimately, the properties of the part. However, there are other elements that can affect the outcome of the final parts such as sintering time, temperature, and atmosphere composition. Recent work has been conducted in this area to understand and to help quantify the positive aspects of sinter hardening.

The goals of this investigation are:

1. To relate the properties of 3 sinter hardenable materials with respect to variations in sintering parameters (sintering time, cooling rate, and atmospheric composition).
2. To gain a quantitative representation of which sinter hardening parameters create the most impact on the final properties of the materials.
3. To better understand the other factors that might influence sinter hardened parts.

PROCEDURE:

3 sinter hardenable materials were analyzed for this investigation:

1. Astaloy A + 2% Cu + 0.8% Graphite + 0.75% Acrawax C
2. D.DH-1 + 0.8% Graphite + 0.75% Acrawax C
3. Astaloy CrM + 0.4% Graphite + 0.75% Acrawax C

Note: materials above made up of following compositions
1. Pre-alloyed (2% Ni + 0.5% Mo)
2. Diffusion bonded (2% Cu + 1.5% Mo)
3. Pre-alloyed (3% Cr + 0.5% Mo)
Each material was pressed into transverse rupture strength bars (TRS) and tensile bars to 7.0 g/cm³.

The TRS and tensile bars were sintered on a six-inch laboratory designed furnace with a convection cooling system. Four (4) temperature profiles were taken to document the cooling rates. It was assumed that the cooling rates for the 30 minutes sintering time would be the same as those obtained for the 10 minutes. The only difference between these cooling rates was the longer holding time in the hot zone from 10 minutes to 30 minutes as an index system was used with the same 6 inch per minute belt speed for the remaining portion of the sintering cycle.

Sintering was performed varying the cooling fan speed from 0 Hz to 10 Hz, H₂ % from 5% to 20%, and sintering time in the hot zone from 10 minutes to 30 minutes. Thus, each material experienced 8 profiles in the sintering furnace.

After sintering, TRS bars and tensile bars were annealed in air for 1 hour at 380 deg. F. Some TRS bars were also left un-annealed.

Hardness was measured on the un-annealed and annealed TRS bars.

TRS was measured on the un-annealed and annealed TRS bars.

Tensile strength and elongation were measured on the annealed tensile bars.

As sintered bars were analyzed at each condition for combined carbon. The Cr based material was also checked for O₂ and N₂ after sintering.

**RESULTS:**

Below is a table representing the time to cool in seconds for the temperature ranges indicated. The data is presented from the fastest cooling rate (CR1) to the slowest cooling rate (CR4). Cooling rate 1 (CR1) was for the condition 20% hydrogen and fan speed set to 10Hz. CR2 through CR4 are as indicated in the table for their conditions. The values are cumulative; i.e. time to cool from 1400 to 400 for CR1 is 162 seconds.

<table>
<thead>
<tr>
<th>Temp (F)</th>
<th>20% H 10 Hz</th>
<th>5% H 10 Hz</th>
<th>20% H 0 Hz</th>
<th>5% H 0 Hz</th>
</tr>
</thead>
<tbody>
<tr>
<td>1400-1300</td>
<td>18</td>
<td>18</td>
<td>18</td>
<td>21</td>
</tr>
<tr>
<td>1300-1200</td>
<td>33</td>
<td>36</td>
<td>33</td>
<td>39</td>
</tr>
<tr>
<td>1200-1100</td>
<td>48</td>
<td>48</td>
<td>48</td>
<td>60</td>
</tr>
<tr>
<td>1100-1000</td>
<td>57</td>
<td>60</td>
<td>63</td>
<td>78</td>
</tr>
<tr>
<td>1000-900</td>
<td>66</td>
<td>69</td>
<td>78</td>
<td>99</td>
</tr>
<tr>
<td>900-800</td>
<td>81</td>
<td>87</td>
<td>99</td>
<td>123</td>
</tr>
<tr>
<td>800-700</td>
<td>102</td>
<td>108</td>
<td>126</td>
<td>150</td>
</tr>
<tr>
<td>700-600</td>
<td>120</td>
<td>129</td>
<td>156</td>
<td>186</td>
</tr>
<tr>
<td>600-500</td>
<td>138</td>
<td>147</td>
<td>201</td>
<td>240</td>
</tr>
<tr>
<td>500-400</td>
<td>162</td>
<td>174</td>
<td>264</td>
<td>315</td>
</tr>
<tr>
<td>C/sec</td>
<td>3.4</td>
<td>3.2</td>
<td>2.1</td>
<td>1.8</td>
</tr>
<tr>
<td>F/min</td>
<td>370</td>
<td>345</td>
<td>227</td>
<td>190</td>
</tr>
</tbody>
</table>

Cooling Rates in degrees C per second and degrees F per minute
A design of experiments (DOE) was conducted to see what were the major contributing factors [1].

From the DOE as well as the raw data, charts were created for each material to demonstrate the effect of the sintering process variations.

Figures 1 and 2 demonstrate the data obtained for the Astaloy A + 2% Cu + 0.8% C mix.

It is seen in Figure 1 how the hardness increases slightly with cooling speed. It is difficult to draw conclusions on any effects on TRS. Figure 2 shows much of the same trends.

Figures 3 and 4 represent the results of the D.DH-1 + 0.8% C mix.
Both Figures 3 and 4 demonstrate the increase of hardness with increasing cooling speed. TRS is affected by the longer sintering time. The greater slope of the hardness curves in relation to increasing cooling speed supports this. This is evidenced by the upward shift of the TRS curves with respect to increasing sintering time.

Figures 5 and 6 show the data from the Astaloy CrM + 0.4% C mix.

Figures 5 and 6 both show how hardness increases with increasing cooling speed. TRS increases with both sintering time and cooling speed.
Total carbon after sintering was conducted on all materials as well as oxygen and nitrogen for the Astaloy CrM grade to verify that the atmosphere was not causing any carburizing, de-carburizing, or oxidizing conditions. The tests were encouraging as changes in carbon were little, only 0.1% less than the as-mixed condition. Oxygen and nitrogen levels were extremely low.

Sintered densities were also measured for each of the materials. Astaloy A mix was 6.83 g/cc; D.DH-1 mix was 6.87 g/cc; and Astaloy CrM mix was 6.93 g/cc.

**DISCUSSION:**

Referencing Table 1 from above, several statements can be made regarding cooling rate. First is that convection cooling is necessary to increase cooling rate. The next statement is that increasing the hydrogen percent increases the cooling rate regardless of the fan speed.

**Ast A Mix:** Some trends have been seen based on the results. For the Astaloy A material, the hardness slightly increased with the increase in the cooling fan speed. Any effects of any sintering process variables on TRS were hard to decipher. It was difficult to show any type of contrast due to processing effects with this material due to the high carbon content and presence of large amounts of martensite. Due to the higher hardness and levels of martensite, the elongation and TRS as well as tensile strength were lower for this material than both of the others.

It is also evidenced in Figure 1 how TRS decreases at the 30 minutes sintering time when the cooling rate increases. More investigation will be required to examine this phenomenon, but the high levels of martensite could in fact cause residual stresses thus decreasing ultimate tensile strength.

**D.DH-1 Mix:** The D.DH-1 material showed some interesting traits due to sintering process variations. The hardness in the tempered state was nearly as high as the Astaloy A material. The cooling rate was the driving force in the hardenability of this material. However, this was not the only finding.

Increased sintering time allows for better sintering necks. It also allows for the copper to diffuse better into the matrix. Figure 7 shows poor copper diffusion at only 10 minutes sintering time. The copper is still present in the bainitic regions.

![Cu-rich bainite](image)

**Figure 7: D.DH-1 + 0.8% C (1% Nital/4% Picral)**

Increasing sintering time from 10 to 30 minutes leads to higher strength due to better sintering necks and copper diffusion. This results in higher TRS and tensile strength values. It was even noticed that even
when the cooling rate decreased along with hardness as well as martensite content, the TRS and tensile strength still increased for the D.DH-1 as long as the sintering time was increasing.

**Ast CrM Mix:** The Astaloy CrM material benefited from both the cooling rate and the sintering time. Hardness as well as TRS were improved from the faster cooling rates and the sintering time yielded better TRS and tensile strength. In this case, cooling rate increases the hardenability and strength of this material. Increased sintering time allows for better sintering.

Table 2 below, shows the percent martensite with respect to sintering time and cooling rate for each material. As mentioned previously, the Astaloy A material had very large amounts of martensite. All materials benefited from the increase in sintering time at each cooling rate, cooling rate one (CR1) being the fastest. D.DH-1 was the only material to have less martensite present with the increase in sintering time for the two fastest cooling rates. The differences in amounts of martensite are minimal though. Some differences in the part location in the sintering furnace could contribute to this. The Astaloy CrM material shows the most contrast in martensitic content, but only 0.4% C was used in this grade.

<table>
<thead>
<tr>
<th>Matl.</th>
<th>Ast A</th>
<th>Ast A</th>
<th>D.DH-1</th>
<th>D.DH-1</th>
<th>Ast CrM</th>
<th>Ast CrM</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>10</td>
<td>30</td>
<td>10</td>
<td>30</td>
<td>10</td>
<td>30</td>
</tr>
<tr>
<td>CR1</td>
<td>98</td>
<td>99</td>
<td>90</td>
<td>85</td>
<td>70</td>
<td>85</td>
</tr>
<tr>
<td>CR2</td>
<td>98</td>
<td>99</td>
<td>90</td>
<td>85</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td>CR3</td>
<td>95</td>
<td>97</td>
<td>55</td>
<td>65</td>
<td>40</td>
<td>40</td>
</tr>
<tr>
<td>CR4</td>
<td>95</td>
<td>99</td>
<td>55</td>
<td>65</td>
<td>25</td>
<td>40</td>
</tr>
</tbody>
</table>

Below are some of the microstuctures seen representing the sintering process variable effects on the materials and their properties.

Figures 8 and 9 represent the Astaloy mix tested at the CR3 and CR2 with 10 minutes sintering time, respectively. 95% martensite/5% bainite (95M/5B) is present in Figure 8, 98M/2B for Figure 9.

![Figure 8: Ast A + 2% Cu +0.8% C (1% Nital/4% Picral) 95M/5B](image-url)
Figure 9: Ast A + 2% Cu + 0.8% C (1% Nital/4% Picral) 99M/1B

Figures 10 and 11 show CR2 and CR4 for the D.DH-1 mix at 10 minutes and 30 minutes sintering, respectively. These represent the decrease in hardness and martensite content, but increase in strength.

Figure 10: D.DH-1 + 0.8% C (1% Nital/4% Picral) 90M/10B
Figures 12 and 13 show the microstructures of the Ast CrM mix. CR1 and CR2 are shown respectively. The only difference between the cooling rates here is the hydrogen content. Increasing the hydrogen for 5% to 20% changed the amount of martensite from 60% to 85% with the balance being bainite.
CONCLUSIONS:

The above investigation revealed some findings. Hardness is primarily dependant on cooling rate. Sintering time had its biggest effect on strength. Hydrogen improved the cooling rate after sintering. Most importantly, the effect of process variables is entirely material dependant. The results of this investigation support this.

The properties and structure of materials is also material dependant. Application details should be addressed before choosing a sinter hardenable grade material. What properties are most desirable for the application? So much emphasis is on hardness when sinter hardening is discussed. Not every application requires the highest hardness. Once application details are discussed, then the correct sinter hardening material and process parameters can be utilized.
REFERENCES: