Production Experience with High Consistency FC-0208 Material Made Using Advanced Bonding Technology

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Abstract
Iron-copper-carbon steels are vital to the PM industry due to their attractive combination of low cost and high performance. However, they often experience instability of dimensional change through the sintering process. This often requires additional sizing and machining operations in order to qualify final part dimensions. In the worst case, this unpredictability can lead to scrapping the as-sintered component, resulting in significant cost implications. This paper is a follow-up study on the efforts to improve dimensional stability of a VVT stator made using FC-0208 material via the use of a binder treated premix and a -15 micron copper powder additive. This paper will present the results of ~20 consecutive truckloads of material demonstrating a significantly reduced dimensional change (DC) variability, which translated into reduced scrappage and improved productivity. Additional studies have focused on the potential causes of sintered dimensional variability in copper steels and how this unique combination of the raw materials, premix processing, and component production have led to the improvements observed.

Introduction
Early in the development of PM ferrous materials, the choice of alloying elements was dictated by the accepted rule: “oxides of alloying elements for mixing with iron powder must be reduced as easily, or more easily, than iron itself” [1]. Copper and graphite additions were chosen because the graphite reacts to form steel and the copper addition contributes to strength and promotes good sintering response. Despite more than 60 years of PM alloy development, the iron-copper-carbon steels are still the pre-eminent material of choice for the majority of automotive PM applications [2]. One very significant change in recent years is the desire to utilize the FC-02XX family of materials in applications requiring
greater mechanical strength and greater dimensional precision [3]. It is this greater dimensional precision that is often in conflict with the basic characteristics of iron-copper steels. What is needed is an iron-copper-carbon system that facilitates improved productivity and dimensional precision with the ease of processing inherent with the FC-020XX material family.

Sintered dimensional change (DC) of PM copper steels is influenced by the amount and type of premixed copper, the amount of graphite in the premix, green part density, and sintering conditions [4, 5]. Growth during sintering results from the melting of copper and subsequent copper diffusion into the iron matrix through both inter-particle surfaces and grain boundaries [6]. Figure 1 illustrates the onset of melting of a ~125 micron copper particle, exhibiting the initial copper particle and grain boundary wetting in an FC-02XX material. Sintering at conventional temperatures will result in complete melting of the copper; however, despite the initial melting and wetting of the iron with copper, complete copper homogeneity is not achieved at conventional sintering temperatures. This results in a non-uniform concentration of copper with copper-rich regions at the prior copper-iron interfaces, creating significant copper gradients within the PM part [6]. The resulting sintered DC will be affected by this copper inhomogeneity. Increasing graphite additions up to 1% reduce the sintered DC by decreasing solid phase grain boundary diffusion of copper into the iron [7, 8].

As reported previously by Shah, improved sintered DC response of FC-0208 materials was realized by utilizing a fine copper addition (~15 micron) in combination with chemical bonding of the premix additives [9]. This synergy of premix processing and alloying selection optimized sintered DC control for a variable valve timing (VVT) stator. The fine copper addition showed two benefits. First, proper dispersion of the finer copper eliminates the large voids that result from the melting of ‘large’ copper particles such as those seen in Figure 1. Secondly, chemical bonding of the fine premix additives ensures that the premix homogeneity achieved during the premixing operation is maintained through powder transport and, ultimately, delivery into the die cavity.

One additional key observation from Shah was the concept of sintered difference from standard (DFS) as the metric to evaluate stability of sintered DC from lot-to-lot. Implicit in this difference from standard evaluation is the prudent choice of the proper standard. Ideally, the standard chosen should represent the mid-point of the dimensional specification, thus enabling a normal distribution of data about the mean. Another key was the use of DFS in addition to absolute DC to rationalize the inherent differences in sintering furnaces between the raw material supplier and the PM part producer.
This paper will detail the experimental methods used to achieve superior dimensional change consistency in an FC-0208 premix used in a VVT application. Part functionality required a +/- 40 micron tolerance on a 3.307 inch (84 mm) diameter. To achieve this level of dimensional precision, the part required sizing after sintering and the critical pump surfaces were ground to tolerance after induction hardening. Minor variations in DC were counteracted by adjusting both the sintering temperature and time at temperature. However, excessive variations could not be tolerated because it required substantial machining or, in the worst case, producing a part that did not meet print specifications. Both instances had significant negative cost implications. To address this issue, a study was undertaken to investigate the potential cause(s) of the variations, what could be done to minimize these variations on a short term basis, and, most importantly, what could be done to ensure long-term stability of the process while maximizing productivity.

Experimental Procedure

A. Laboratory Studies

The initial experimental work performed investigated the effects of copper addition type and premixing alternatives. In this phase of the study, four 500 pound (227 kg) premixes were prepared as detailed in Table 1. In all premixes, the base iron utilized was Hoeganaes Corporation Ancorsteel 1000C, the carbon addition was 0.72% natural graphite, and the lubricant addition was 0.75% EBS. Once the laboratory-sized premixes were prepared, they were evaluated for basic powder properties of apparent density and flow, compressibility, sintered dimensional change, and sintered TR strength [10]. One additional test performed on each premix was elutriation to measure the potential dusting resistance of each premix [11]. This test uses a steady flow of nitrogen gas that fluidizes a column of powder with the objective to segregate the low density or small particle size premix additives. High dusting resistance implies a reduced tendency to segregate during transport and subsequent powder handling during PM part production.

Table 1
Initial premixes evaluating the effects of copper type and premixing alternative

<table>
<thead>
<tr>
<th>Premixing alternative</th>
<th>Copper type</th>
<th>% Copper type addition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard premix</td>
<td>-150 micron</td>
<td>1.70</td>
</tr>
<tr>
<td>Standard premix</td>
<td>Diffusion bonded 20% copper master alloy</td>
<td>8.50 (1.70 total copper)</td>
</tr>
<tr>
<td>Ancorbonded</td>
<td>-150 micron</td>
<td>1.70</td>
</tr>
<tr>
<td>Ancorbonded</td>
<td>-15 micron</td>
<td>1.70</td>
</tr>
</tbody>
</table>

B. Production Testing

Figure 2 shows the part investigated in this study. This VVT part had three levels with a major sprocket diameter of ~5.3 inches (134.6 mm), an inner diameter of 3.307 inches (84 mm), and an overall height of ~0.8 inches (20 mm). Part mechanical requirements necessitated that the sprocket flange region maintain a sintered density of ~6.9 g/cm³, while the specification of the major long hub (Figure 2B) was an overall green density of ~6.8 g/cm³. The major short hub is formed by a fixed step in the upper punch (Figure 2A). Compaction was performed utilizing a mechanical press and sintering was done nominally at 2050
°F (1120 °C) for ~25 minutes at temperature in a 95 vol% nitrogen / 5 vol% hydrogen atmosphere. All material used in production was an MPIF FC-0208 powder produced via Hoeganaes’ proprietary ANCORBOND® processing. Quality control testing of the premix evaluated each premix lot for sintered carbon, sintered copper, absolute DC, and DC as measured via difference from a standard lot sintered simultaneously with the production lot. All dimensional change data was measured using MPIF standard TRS bars compacted to a 7.0 g/cm³ green density and sintered at 2050 °F (1120 °C) in a 75 vol% hydrogen / 25 vol% nitrogen atmosphere for 30 minutes at temperature. During the course of this study, approximately 20 lots of material were evaluated, representing greater than 800,000 pounds (363,000 kg) of supplied material, or approximately six months of actual part production. Additional production testing assessed the weight uniformity of as compacted components by measuring 30 consecutive parts for each of two lots twice a day for three days of production.

Figure 2: Photograph of VVT stator showing major short hub OD (A) and major long hub (B)

Results

A. Laboratory Studies—Copper Type and Premix Alternatives

Table 2
AD & Flow of Laboratory Prepared Premixes

<table>
<thead>
<tr>
<th>Mix</th>
<th>Apparent Density (g/cm³)</th>
<th>Flow (s/50g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Regular Copper, Standard premix</td>
<td>2.95</td>
<td>31</td>
</tr>
<tr>
<td>Diffusion Alloyed Copper, Standard premix</td>
<td>2.94</td>
<td>31</td>
</tr>
<tr>
<td>Regular Copper, Ancorbonded</td>
<td>3.05</td>
<td>28</td>
</tr>
<tr>
<td>Fine Copper, Ancorbonded</td>
<td>3.21</td>
<td>27</td>
</tr>
</tbody>
</table>
Table 2 presents the measured apparent density (AD) and flow of the four mixes evaluated. Conventional double cone blending of the standard copper and the diffusion bonded copper addition gave nearly identical AD and flow. Chemical bonding of the standard copper increased the AD by approximately 0.1 g/cm³ with a 10% improvement in flow. Similarly, chemical bonding of the -15 micron copper powder increased the AD to ~3.2 g/cm³ with additional improvement in the flow. The higher AD lowers the fill required to produce a part and the improved flow opens the opportunity to increase press speed with no degradation of quality.

Elutriation values presented in Figure 3 demonstrate two trends. First, graphite is more susceptible to dusting relative to copper. Graphite’s density is 2.2 g/cm³ and the fine particle size of the additive does promote segregation during the processing of the premix and, ultimately, the PM part. Copper has a density of approximately 8.1 g/cm³, which is nearly the same as iron. This, combined with the relatively coarser particle size distribution of the copper, does minimize the potential for segregation. It is important to note that both carbon and copper variations can result in variations in sintered DC. Thus, the chemical bonding of the graphite is significant to eliminate this potential source of variation. The diffusion bonding of the copper as an alloying addition is not necessary to eliminate potential sources of variation. Dusting resistance of both the standard copper premix and chemically bonded fine copper show nearly identical copper values after completion of the elutriation testing.

Figures 4, 5 and 6 present the sintered dimensional change, sintered TR strength, and sintered apparent hardness for the four laboratory premixes, respectively. As seen in Figure 4, the addition of the -15 micron copper powder promotes greater absolute sintered dimensional change. This results from the greater number of iron-copper particle contacts, thus promoting greater initial copper diffusion during the sintering process with the corresponding greater swelling of the iron lattice. This should not be considered a detriment, provided that within-lot and lot-to-lot consistency of the powder is maintained so as to produce consistent sintering behavior. Varying the particle size of the copper does not significantly affect the as-sintered strength or as-sintered apparent hardness of the FC-0208 premix.
Figure 4:  Dimensional change of various copper additions vs. green density

Figure 5:  TRS of various copper types vs. sintered density
Figures 7 and 8 present the metallographic analysis of test samples prepared from each of the four laboratory premixes in the as polished and etched conditions. Figures 7A, 7C, 8A, and 8C depict the addition of the -150 micron copper powder. As discussed, the melting of the relatively coarse copper does result in the presence of larger pores occurring from the melting and subsequent diffusion of the large copper particles. Figures 7D and 8D depict the addition of the -15 micron copper with the corresponding smaller and more rounded porosity. Figures 7B and 8B are the photomicrographs of the iron premixed with the diffusion alloyed copper master alloy additive. The resulting porosity is intermediate between the coarse and fine copper particle size additions.

Table 3
Axial Fatigue Results

<table>
<thead>
<tr>
<th>Premix</th>
<th>Sintered Density, g/cm³</th>
<th>50% Confidence Limit, psi</th>
<th>90% Confidence Limit, psi</th>
<th>Standard Deviation, psi</th>
</tr>
</thead>
<tbody>
<tr>
<td>Production Premix utilizing -15 Micron Copper</td>
<td>6.93</td>
<td>18,500</td>
<td>16,750</td>
<td>1,290</td>
</tr>
<tr>
<td>Laboratory Premix utilizing -150 Micron Copper</td>
<td>6.91</td>
<td>16,650</td>
<td>15,050</td>
<td>1,170</td>
</tr>
</tbody>
</table>
Figure 7: As polished microstructures for regular copper premix (A), diffusion alloyed premix (B), bonded regular copper (C), and bonded fine copper (D)

The significance of the smaller pore sizes associated with the -15 micron copper premix addition does not manifest itself in the static strength values shown in Figures 5 and 6. However, axial fatigue testing of a production premix utilizing the -15 micron copper vs. the standard -150 mesh copper was performed. Shown as Table 3 is a summary of the axial fatigue testing (R= -1) of specimens compacted to a 7.0 g/cm³ green density. This data suggests that the inherently smaller porosity of the -15 micron copper results in approximately 10% higher fatigue life for both the 50% and 90% confidence limits. All data was calculated via the methodology outlined in MPIF Standard Test Methods, Standard 56 [10].
Additional metallography was performed on a production premix sintered at temperatures of 1037 °C, 1065 °C, 1081 °C, and 1085 °C, with results presented in Figures 9A through 9D, respectively. The rationale for this was to determine how the -15 micron copper diffused into the iron above and below the melting point of copper (1083 °C). As expected at 1037 °C, the copper particles are readily apparent in the photomicrograph (9A) and the interfaces between the copper and iron particles are well defined. Raising the sintering temperature to 1065 °C, the copper particles are still evident in the microstructure, Figure 9B. It appears that the interfaces between the copper and iron particles are less defined, possibly indicating some initial diffusion of the fine copper into the iron. At 1081 °C, the amount of undiffused copper has decreased significantly and the remaining copper particles are in intimate contact with the iron particles, Figure 9C. Lastly, at 1085 °C, the copper is almost 100% diffused into the iron with only minor amounts of undiffused copper, Figure 9D. This study illustrated the greater initial diffusion of the -15 micron copper resulting from the increased number of iron-copper particle contacts. This is in contrast with the larger amount of undiffused copper evident in Figure 1. The greater initial diffusion of the -15 micron copper addition is responsible for the higher growth observed in Figure 4. In addition to better diffusion, because the copper is chemically bonded to the iron, the fine copper will promote reduced segregation in the microstructure.

Figure 8: Etched microstructures for regular copper premix (A), diffusion alloyed premix (B), bonded regular copper (C), and bonded fine copper (D)
B. Production Results

In the prior published work by Shah [9], it was reported that at the inception of this effort, dimensional variations were resulting in unacceptable levels of rejected parts. Pareto analysis showed that the major cause for part rejection was an undersized condition on the critical 84 mm diameter dimension [9]. Initially, premix modifications were enacted to produce greater sintered dimensional change. The original premix was a chemically bonded premix utilizing the -150 micron copper additive. To increase the sintered DC, a combination of regular (-150 micron) and fine (-15 micron) copper was utilized, exploiting the trend shown in Figure 4. Although successful, this approach required lot-to-lot adjustments in the amount of the fine copper addition, so as to produce the desired result. The second and final iteration on the premix evaluated the use of only fine copper to affect the dimensional change desired. This iteration was pursued vigorously because it offered the potential to chemically bond the fine copper, thus preventing potential segregation effects, and it offered the possibility of a slight reduction in the total amount of copper added to achieve the same absolute dimensional change. Outputs from the initial study showed that with proper selection of a testing standard and utilizing chemical bonding with the fine copper significantly reduced the lot-to-lot variation.
In this study, the key characteristics of apparent density, absolute sintered dimensional control, and DC difference from standard were monitored with the objective of determining the resulting part performance. Figure 10 presents the AD for the 20 lots examined in this study; the data shows a total variation of 0.06 g/cm³ over the 20 lots produced. The significance of this tight control of AD is reduced press adjustment between lots as received for production. Previous work has shown that the chemical bonding has excellent consistency within lot. Thus, this results in reduced tooling adjustments leading to improved overall productivity.

Figures 11 and 12 present the absolute dimensional change and the dimensional change DFS data determined at both the premix production facility and at the parts producer. As expected, the absolute DC does vary between the two different sintering locations. However, the overall range of absolute DC is identical from the two locations. DFS testing also showed similarity of results from the two locations. Interestingly, the DFS at both locations showed a total variation of just 0.05% over the 20 lots evaluated; differences existed between the two locations but the lot-to-lot consistency remained at the same level. Implications of this data are reduced set up time as premix material lots are utilized in production, an overall lowering of scrap rates because of reduced changeover, and greater press and sintering furnace utilization because fewer changes are required.
Sintered carbon was measured at the two locations as well and this data is shown in Figure 13. The chemical bonding promotes very consistent sintered carbon results at the two sintering locations. Consistency of sintered carbon is critical to maintain the restrictive DC necessary for this part. Variations in sintered carbon can significantly alter the sintered DC response.

Figure 13: Sintered carbon content measured at two locations
Figure 14 and Table 4 present the consistency of part weights in production utilizing two lots and over three days of production for each lot. Significant in this data is the relatively tight control capability. Specification for the part is a green weight range of 530 to 536 grams. For each run, the consistency observed was approximately 50% of the given specification range. As importantly, over the production cycle for each lot, minimal variation in weight was observed. Shown in Table 4 is a column representing the potential density variation resulting solely from weight variation observed for each measured run. It is noteworthy that the calculated density range ($6\sigma$) was at most 0.04 g/cm³ for the part. This means that the potential DC variation from the max density variation is less than 0.005%, as shown in Figure 15. This illustrates that DC is not just from potential chemical variations but can also arise from variations in green density as well. To maintain the DC control required for a demanding application such as a VVT component, maintaining both rigid chemical control and part density will facilitate the required part performance. The consistency of both material AD and part weight in this study show a capability to maintain tight density control for this application.

Table 4
Density Variations Resulting from Weight Variations in Figure 15

<table>
<thead>
<tr>
<th>Run</th>
<th>Date</th>
<th>Average Weight (g)</th>
<th>Standard Deviation (g)</th>
<th>Corresponding Density Range ($6\sigma$, g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>12/22/2016 AM</td>
<td>534.23</td>
<td>0.4159</td>
<td>0.0318</td>
</tr>
<tr>
<td>2</td>
<td>12/22/2016 PM</td>
<td>533.06</td>
<td>0.3770</td>
<td>0.0288</td>
</tr>
<tr>
<td>3</td>
<td>12/27/2016 AM</td>
<td>532.09</td>
<td>0.3207</td>
<td>0.0245</td>
</tr>
<tr>
<td>4</td>
<td>12/27/2016 PM</td>
<td>533.25</td>
<td>0.5273</td>
<td>0.0403</td>
</tr>
<tr>
<td>5</td>
<td>12/28/2016 AM</td>
<td>533.98</td>
<td>0.5072</td>
<td>0.0388</td>
</tr>
<tr>
<td>6</td>
<td>12/28/2016 PM</td>
<td>533.74</td>
<td>0.4964</td>
<td>0.0379</td>
</tr>
</tbody>
</table>
Discussion:

This study was a follow-up effort to work that was reported at the 2016 PowderMet conference [9]. The initial study focused on the methodology used to reduce the dimensional change variability in an FC-0208 premix used for a VVT application. Through the use of chemical bonding coupled with a -15 micron copper particle addition, a significant reduction in non-conforming parts was observed. Rejection rates fell from a high of about 5% to a current level less than 0.5%. However, in the previous work, the time span of observation was limited to about three weeks. In this study, the time frame was expanded to about six months and the amount of premixed material evaluated was in excess of 800,000 pounds.

In this effort, the laboratory evaluation of various copper additions demonstrated that chemical bonding of the graphite and fine copper promotes high apparent density with improved flow. Significance of this is the reduced die fill depth with high AD and the potential for faster compaction rates because of the reduced fill and improved flow rates. Along with these two advantages, the chemical bonding demonstrated reduced potential for graphite segregation. Segregation of copper was minimal for the premixes evaluated; however, the high shear mixing utilized in the chemical bonding gives rise to enhanced copper dispersion throughout the premix coupled with minimal graphite dusting. These two factors are key to enhancing the dimensional precision of the premix resulting in enhanced production. It was also shown that the -15 micron copper addition promoted higher sintered DC at equivalent green densities when compared to a -150 micron copper particle addition. It should be stressed that although higher, this is not a problem provided that the AD and DC values are consistent within lot and from lot-to-lot. No differences in compressibility or sintered strength were observed. However, the -15 micron copper addition also promotes smaller porosity within the sintered part, with the potential advantage of better dynamic properties, in particular, fatigue.
Production experience with this enhanced premixing alternative demonstrated consistent part performance over a six month time frame. Sintered dimensional change both absolute and difference from standard maintained a total variation of +/- 0.025% over the time frame reported. This is particularly significant in light of the fact that an FC-0208 material was utilized. In addition to the tight sintered DC control realized, the other key variable was the AD of premix. Over the six month range, the total variation of AD was 0.06 g/cm³. This tight control reduced lot-to-lot compaction die set up adjustments, thus increasing productivity. It was also deduced that this tight control of AD also minimized green part weight variations, leading to extremely low green part density variations to a level that at most resulted in a total DC variation of 0.005%.

One factor observed in the laboratory part of this study was the reduced dusting of the graphite via the chemical bonding. Production experience during powder and part production showed the variability of sintered carbon was reduced to less than +/- 0.03%. Again, it is the combination of reduced sintered carbon variability, enhanced copper distribution, with excellent part-to-part weight consistency that enabled the extremely small DC variations observed in this study.

One final point worth noting is the cost implications of this extra powder premix processing. The -15 micron powder has a higher cost to produce than the standard -150 micron material. Additionally, the chemical bonding has a higher charge than standard double cone premixing. But, the cost of the premixed powder is only one consideration in the final part cost. As shown in Figure 16 using a developed cost model, the cost of sintered part scrap as it affects total part cost is significant. Relating to this study, at the ~5% scrap rate initially observed with this component, the extra cost of the fine copper and enhanced premixing is completely offset by the reduction in scrap rates only. It should also be noted that the data present in Figure 16 just relates to part cost without any consideration to factory utilization and additional press availability that may be realized through use of high consistency, precision bonded material.

![Figure 16: Cost Analysis of various scrap rates with standard and -15 micron copper powder](image)
Summary

As a result of the experimental and production work performed during this study, the following observations were made:

1. More than 800,000 pounds of premixed powder was used in the production of a VVT stator having tight dimensional tolerances. Productivity was increased and scrap rates were reduced significantly.
2. This work has demonstrated that it is possible to maintain tight dimensional control of an FC-0208 premix; careful control of the premixing, dust prevention of alloying elements, and tight part weight control is also required.
3. Despite differences in sintering atmospheres and sintering furnaces between the powder mixing facility and the production facility, the tight DC tolerance could be maintained using a DFS criteria, using a carefully selected standard material lot.
4. Using a diffusion alloyed copper source is not necessary for these tight tolerances.
5. Strict control of part green density is important. Excessive variations can result in producing production parts that will not meet part dimensional specifications.
6. The -15 micron copper addition does not show improved transverse rupture strength but does show improved axial fatigue life. This results from the absence of large pores that can result from the addition large copper particles in the standard -150 micron copper.
7. The -15 micron copper particles show substantial diffusion at temperatures below the melting point of copper. This results in a more uniform copper distribution and larger absolute growth upon sintering.
References