PRODUCTION EXPERIENCE WITH HIGH-CONSISTENCY FC-0208 MATERIAL MADE USING ADVANCED BONDING TECHNOLOGY

Suresh Shah*, Gerry Wewers, PMT**, Gregory Falleur, PMTII***, Bridget Reider****, Francis Hanejko***** and Kylan McQuaig******

INTRODUCTION

Early in the development of powder metallurgy (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.” 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. 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 better dimensional precision. 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. 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. Figure 1 illustrates the onset of melting of an approximately 125 micrometre copper particle, exhibiting the initial copper particle and grain boundary wetting in an FC-02XX material. Sintering at conventional temperatures (1,120 °C (2,050 °F)) will result in complete melting of the copper. However, despite the initial melting and wetting of the iron with copper, complete copper homoge-

*Chief Metallurgist, **Powder Metallurgy Technologist, ***Senior Powdered Metal Engineer, American Axle & Manufacturing, Inc.—Powertrain, Subiaco Manufacturing Facility, P. O. Box 81, Hwy 22 & Leo Ave, Subiaco, Arkansas 72865, USA, ****Laboratory Manager, Hoeganaes Corporation, 4330 Paradise Road, Watsontown, Pennsylvania 17777, USA, *****Retired (formerly Manager Customer Applications, Hoeganaes Corporation), ******Global R&D Manager, Hoeganaes Corporation, 1001 Taylors Lane, Cinnaminson, New Jersey 08077, USA; Email: kylan.mcquaig@hoeganaes.com

neity 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. The resulting sintered DC will be affected by this copper inhomogeneity. Increasing the graphite addition to 1 wt.% reduces the sintered DC by decreasing solid phase grain-boundary diffusion of copper into the iron.

As reported previously by Shah, improved sintered DC response of FC-0208 materials was realized by utilizing a fine copper addition (minus 15 μm) in combination with binder treatment of the premix additives. 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. Proper dispersion of the finer copper eliminates the large voids that result from the melting of ‘large’ copper particles such as seen in Figure 1. In addition, binder treatment 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 reference (DFR) as the metric to evaluate the stability of sintered DC from lot-to-lot. Implicit in this difference from reference evaluation is the prudent choice of the proper reference material. Ideally, the reference 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 DFR 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 μm tolerance on an 84-mm (3.307-inch) 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 they required substantial machining or, in the worst case, production of 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**

**Laboratory Studies**

The initial experimental work performed investigated the effects of copper addition type and premixing alternatives. In this phase of the study, four 225 kg (500 lb) premixes were prepared as detailed in Table I. In all premixes, the base iron utilized was Hoeganaes Corporation Ancorsteel 1000C, the carbon addition was 0.72 wt.% natural graphite, and the lubricant addition was 0.75 wt.% 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.

<table>
<thead>
<tr>
<th>Premixing Alternative</th>
<th>Copper Type</th>
<th>Copper Type Addition (wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional premix</td>
<td>minus 150 μm</td>
<td>1.0</td>
</tr>
<tr>
<td>Conventional premix</td>
<td>diffusion-alloyed 20 wt.% copper master alloy</td>
<td>8.5 (1.7 total copper)</td>
</tr>
<tr>
<td>Binder-Treated Premix</td>
<td>minus 150 μm</td>
<td>1.7</td>
</tr>
<tr>
<td>Binder-Treated Premix</td>
<td>minus 15 μm</td>
<td>1.7</td>
</tr>
</tbody>
</table>

Figure 1. Copper particle at the onset of melting (1,083 °C (1,981 °F))

Figure 2 shows the part investigated in this study.
This VVT part had three levels with a major sprocket diameter of 134.6 mm (~5.3 inches), an inner diameter of 84 mm (3.307 inches), and an overall height of 20 mm (~0.8 inches). 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 2(b)) 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 2(a)). Compaction was performed utilizing a mechanical press and sintering was done nominally at 1,120 ºC (2,050 °F) 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 reference lot sintered simultaneously with the production lot. All dimensional change data were measured using MPIF standard TRS bars compacted to a 7.0 g/cm³ green density and sintered at 1,120 ºC (2,050 °F) 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 363,000 kg (800,000 lb) 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.

**RESULTS**

*Laboratory Studies—Copper Type and Premix Alternatives*

Table II presents the measured apparent density (AD) and flow of the four mixes evaluated. Conventional double-cone mixing of the standard copper and the diffusion-alloyed copper addition gave nearly identical AD and flow. Binder treatment of the standard copper increased the AD by approximately 0.1 g/cm³ with a 10% improvement in flow. Similarly, binder treatment of the minus 15 μm 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

![Figure 2. Photograph of VVT stator showing major short hub OD (a) and major long hub (b)](image)

![Figure 3. Elutriation of carbon and copper of the four laboratory premixes](image)
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 2.3 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 8.9 g/cm³, which is slightly higher than iron. This, combined with the relatively coarser particle-size distribution of the copper, does minimize the potential for dusting. It is important to note that both carbon and copper variations can result in variations in sintered DC. Thus, the binder treatment of the graphite is significant to eliminate this potential source of variation. The diffusion-alloying of the copper is not necessary to eliminate potential sources of variation. Dusting resistance of both the standard copper premix and binder-treated 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 respectively for the four laboratory premixes. As seen in Figure 4, the addition of the minus 15 μm 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 within-lot and lot-to-lot consistency of the powder is maintained 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.

Figures 7 and 8 present the metallographic analysis of test samples prepared from each of the four laboratory premixes in the as-polished condition and after etching respectively. Figures 7(a), 7(c), 8(a), and 8(c) depict the addition of the minus 150 μm 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 7(d) and 8(d) depict the addition of the minus 15 μm copper with the corresponding smaller and less irregularly shaped (smoothed) porosity. Figures 7(b) and 8(b) 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.

The significance of the smaller pore sizes associated with the minus 15 μm 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 minus 15 μm copper vs. the standard minus 150 μm copper was performed. Table III is a summary of the axial fatigue testing (R = -1) data for specimens compacted to a 7.0 g/cm³ green density. The test bars conformed to ISO 3928 (90-mm long, 11-mm wide at the ends and 5-mm wide
PRODUCTION EXPERIENCE WITH HIGH-CONSISTENCY FC-0208 MATERIAL MADE USING ADVANCED BONDING TECHNOLOGY

TABLE III. 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 minus 15 μm copper</td>
<td>6.93</td>
<td>18,500</td>
<td>16,750</td>
<td>1,290</td>
</tr>
<tr>
<td>Laboratory Premix utilizing minus 150 μm 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 (a) regular copper premix, (b) diffusion-alloyed premix, (c) bonded regular copper, and (d) bonded fine copper.

at the central section with a 30-mm radius—the bars were 6-mm thick). These data suggest that the inherently smaller porosity of the minus 15 μm copper results in approximately 10% higher fatigue life. All data were calculated via the staircase methodology outlined in MPIF Standard Test Methods, Standard 56.10

Additional metallography was performed on a production premix sintered at temperatures of 1,037 °C, 1,065 °C, 1,081 °C, and 1,085 °C (1,899 ºF, 1,949 ºF, 1,978 ºF, and 1,985 ºF respectively) with results presented in Figures 9(a) through 9(d), respectively. The rationale for this was to determine how the minus 15 μm copper diffused into the iron above and below the melting point of copper (1,083 °C (1,981 ºF)). As expected at 1,037 °C (1,899 ºF), the copper particles are readily apparent in the photomicrograph (9(a)) and the interfaces between the copper and iron particles are well defined. After raising the sintering temperature to 1,065 °C (1,949 ºF), the copper particles are still evident in the microstructure, Figure 9(b). 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 1,081 °C (1,978 ºF), the amount of undiffused copper has decreased significantly and the remaining copper particles are in intimate contact with the iron particles, Figure 9(c). Lastly, at
1,085 °C (1,985 °F), the copper is almost 100% diffused into the iron with only minor amounts of undiffused copper, Figure 9(d). This study illustrated the greater initial diffusion of the minus 15 μm 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 minus 15 μm copper addition is responsible for the higher growth observed in Figure 4. In addition to better diffusion, because the copper is bonded to the iron, the fine copper will promote reduced segregation in the microstructure.

**Production Results**

In prior published work by Shah, 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. Initially, premix modifications were enacted to produce greater sintered dimensional change. The original premix was a binder-treated premix utilizing the minus 150 μm copper additive. To increase the sintered DC, a combination of regular (minus 150 μm) and fine (minus 15 μm) 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 of the premix evaluated the use of only fine copper to affect the desired dimensional change. This iteration was pursued vigorously because it offered the potential to binder treat 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 reference and utilizing binder treatment 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 reference were monitored with the objective of determining the resulting part performance.

**Figure 8. Etched microstructures for (a) regular copper premix, (b) diffusion-alloyed premix, (c) bonded regular copper, and (d) bonded fine copper**
Figure 10 presents the AD for the 20 lots examined in this study; the data show 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 received for production. Previous work has shown that binder-treated premixes have excellent within-lot consistency. This results in reduced tooling adjustments and improved productivity.

Figures 11 and 12 present the absolute dimensional change and the dimensional change DFR data deter-
mined 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. DFR testing also showed similarity of results from the two locations. Interestingly, the DFR 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 these 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 these data are shown in Figure 13. The binder treatment 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 14 and Table IV present the consistency of part weight in production utilizing two lots and over three days of production for each lot. Significant in these data is the relatively tight control capability. The 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 important, over the production cycle for each lot, minimal variation in weight was observed. A column in Table IV represents the potential density variation resulting solely from the weight variation observed for each measured run. It is noteworthy that the calculated density range (σ) 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 related to potential chemical variations but can also arise from variations in green density. To maintain the DC control required for a demanding application such as a VVT component, maintaining both rigid chemical composition 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>
<thead>
<tr>
<th>Run</th>
<th>Date</th>
<th>Average Weight (g)</th>
<th>Standard Deviation (g)</th>
<th>Corresponding Density Range (6σ, g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>12/22/2016 AM</td>
<td>534.23</td>
<td>0.42</td>
<td>0.03</td>
</tr>
<tr>
<td>2</td>
<td>12/22/2016 PM</td>
<td>533.06</td>
<td>0.38</td>
<td>0.03</td>
</tr>
<tr>
<td>3</td>
<td>12/27/2016 AM</td>
<td>532.09</td>
<td>0.32</td>
<td>0.03</td>
</tr>
<tr>
<td>4</td>
<td>12/27/2016 PM</td>
<td>533.25</td>
<td>0.53</td>
<td>0.04</td>
</tr>
<tr>
<td>5</td>
<td>12/28/2016 AM</td>
<td>533.98</td>
<td>0.51</td>
<td>0.04</td>
</tr>
<tr>
<td>6</td>
<td>12/28/2016 PM</td>
<td>533.74</td>
<td>0.50</td>
<td>0.04</td>
</tr>
</tbody>
</table>
DISCUSSION

This study was a follow-up effort to work that was reported at POWDERMET2016. 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 binder treatment coupled with a minus 15 μm 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 363,000 kg (800,000 lb).

In this effort, the laboratory evaluation of various copper additions demonstrated that binder treatment of the graphite and fine copper promotes high apparent density with improved flow. The 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 binder-treated premixes demonstrated reduced potential for graphite segregation. Segregation of copper was minimal for the premixes evaluated. However, the high-shear mixing utilized in the binder-treatment process gives rise to enhanced copper dispersion and uniformity 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 minus 15 μm copper addition promoted higher sintered DC at equivalent green densities when compared with a minus 150 μm 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 minus 15 μm copper addition also promotes smaller pores 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 reference 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 binder-treatment processing. 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 minus 15 μm powder has a higher cost to produce than the standard minus 150 μm material. Additionally, the binder treatment has a higher charge than regular double-cone premixing. But, the cost of the premixed pow-
der 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 relate 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.

SUMMARY

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

1. More than 363,000 kg (800,000 lb) 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 DFR criteria, using a carefully selected reference 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 minus 15 μm 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 minus 150 μm copper.

7. The minus 15 μm 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