

Dimensional Precision of Fe-Cu-C Premixes

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Abstract

Iron-copper-carbon powder metallurgy steels are the most widely used materials in the PM industry because of their ease of processing, good mechanical properties and relatively low cost. Dimensional precision with these steels is a challenge, however, and is driven by the growth associated with copper. Admixed copper can segregate in the powder mixture and lead to significant variation in part sizes. The use of these steels in components with tight dimensional requirements, including VVTs, has driven powder producers to develop alternative alloying and processing methods to reduce the dimensional variation. This paper outlines the dimensional response of a standard Fe-Cu-C premix and improvements that have been made to control variation.

Keywords - dimensional precision; VVT; Fe-Cu-C

Introduction

Copper steels are the backbone material of the PM industry. They are used in nearly every automotive mega program with their overall usage surpassing other material systems used in powder metallurgy (PM). [1] This is not to imply that other alloy systems are not important; however, the versatility and cost-effectiveness of iron-copper-carbon steels make them the alloy system of choice for many applications. One potential drawback of the iron-copper-carbon alloy system is the variability of the dimensional control from lot-to-lot and occasionally within lot of premixes. This often requires the need for secondary processing such as sizing or machining to control critical dimensions. A prime example of an automotive component having tight dimensional control is VVT components; they often require holding less than $\pm 0.04\%$ DC on the critical pump dimensions. [2]

Sintered dimensional change 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. [3, 4] The sintered growth results from the copper melting and subsequent copper diffusion into the iron through both particle and grain boundaries. [5] Figure 1 is a photomicrograph of a ~ 125 micron copper particle at the onset of copper melting (1083°C) exhibiting the initial particle and grain boundary wetting. The copper particle shown in Figure 1 results in a non-uniform concentration of copper and relies on diffusion to homogenize the copper distribution. Conventional sintering times and temperatures will not produce a homogeneous copper distribution throughout the microstructure of the iron compact. [6] Increasing graphite additions up to 1% reduce the sintered DC by decreasing solid phase grain boundary diffusion of copper into the iron. [7, 8]

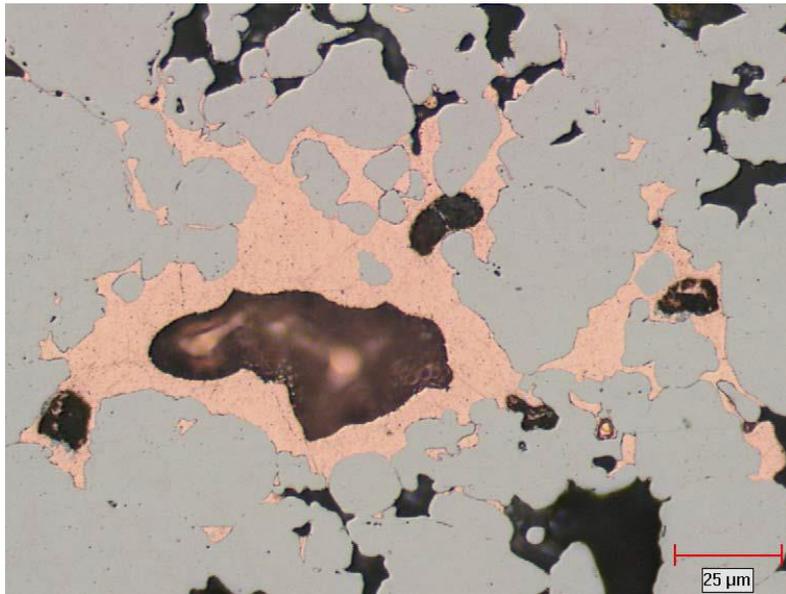


Figure 1: Copper particle at the onset of melting (1083°C), [6].

The majority of copper powders used in PM has a ~ 150 micron particle size distribution; thus, when the large copper particles melt, they leave large porosity within the microstructure. It has been suggested that smaller copper particles (~ 15 microns) are potentially superior because these smaller copper particle sizes minimize / eliminate large pores resulting from copper melting. Shown in

Figure 2 is an FC-0208 material that utilized a -15 micron particle size copper. The greater number of copper particles is dispersed uniformly throughout the structure and upon melting will exhibit improved copper homogeneity. Figure 3a and 3b show the resulting sintered pore morphology when using a -150 micron copper powder and a -15 micron copper powder. Assuming a -150 micron particle size has a D50 of ~35 microns, then a 2% addition has approximately 10 million copper particles per 100 grams of a FC-0208 premix. If a -15 micron particle size is used (assuming a D50 of 11 microns) the number of copper particles per 100 grams has increased to ~300 million. This greater number of iron-copper particle interfaces enhances copper diffusion into the iron prior to the copper melting. [3, 6, 10] A critical requirement for the reduced copper particle size is that they are uniformly dispersed throughout the premix. One methodology to prevent the fine copper from segregating is to 'bond' the copper to the iron. Diffusion bonding and chemical bonding prevent copper segregation but diffusion bonding adds additional cost to the raw material. Chemical bonding has proven effective at bonding these fine copper particles. [11] Ideally it should be advantageous to prealloy the copper in the iron powder. However, prealloying this amount of copper will have a deleterious effect on the compressibility of the resulting iron powder.

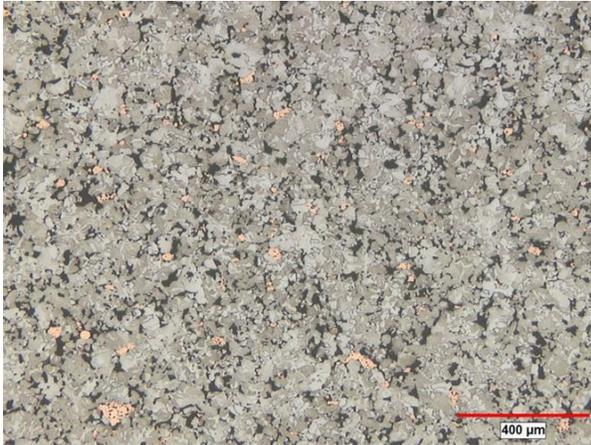
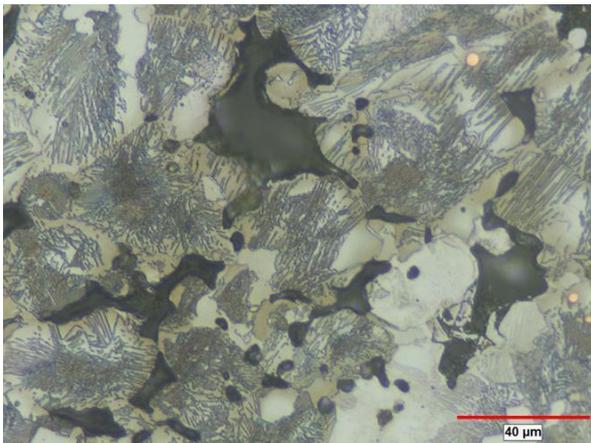
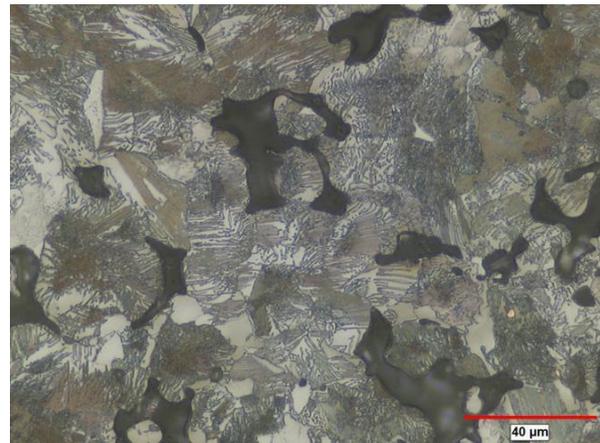


Figure 2: Photomicrograph showing the distribution of -15 micron copper particles in FC-0208

Other considerations in the dimensional control of copper steels are the effect of varying part density, sintering atmosphere and particle oxides present on the powder surfaces. Higher part densities result in higher DC; for example, an FC-0208 at 6.8 g/cm³ green density will have a sintered DC of ~0.3%; whereas, at a green density of 7.3 g/cm³ the sintered DC will be ~0.5%. [3] This presents two problems; parts having multi-levels with varying density distributions require careful die design to accommodate the significant difference in absolute dimensional change. Additionally, achieving high sintered component density (>7.2 g/cm³) in copper steels requires the use of diffusion bonded copper additions to minimize growth at these high green densities. [3] Relative to sintering atmospheres and particle oxides; Bernardo and others demonstrated that a reducing atmosphere significantly lowers the contact angle of the copper during sintering. [5] Also, if copper powders are pre-alloyed with 2% silicon, these additions dramatically lowers the wetting angle in both inert and nitrogen hydrogen atmospheres. The silicon is thought to act as a type of 'getter' to help reduce the iron powder surface oxides.



3a: With -150 micron copper



3b: With -15 micron copper

Figure 3: Evidence of large pores from -150 micron copper and finer pores from a finer copper particle

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 3.3 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 excessive machining or, in the worst case, producing a part that was out of specification. Both instances resulted in significant negative cost implications. To address this issue, a study was undertaken to investigate the potential cause 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 minimizing rejections.

Experimental Procedure

The VVT part investigated was a three-level part having a major sprocket diameter of ~5.3 inches (134.6 mm) with an inner diameter of 3.3 inches (84 mm) and an overall height of ~0.8 inches (20 mm), see Figure 4. 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 was an overall green density of ~6.8 g/cm³ min. The major short hub is formed by a fixed step in the upper punch. 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% nitrogen / 5% hydrogen atmosphere. The MPIF FC-0208 powder was premixed using 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 (DFS) 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% hydrogen / 25% nitrogen atmosphere for 30 minutes at temperature.

Critical part dimensions on this VVT component are the major ID short hub OD and long hub OD as shown in Figure 4. As noted earlier, both required a sintered diametrical tolerance of +/- 40 microns. To establish the required sintering parameters, sintering trials of an initial quantity of parts were done from each new lot quantifying the required temperature and time at temperature. However, only minor variations in these two parameters were permitted. Excessive variations resulted in parts that could not be properly sized.



Figure 4: Photograph of VVT part showing major short hub OD on left and major long hub on right

Results

At the inception of this program, dimensional variations were resulting in unacceptable levels of rejected parts. Pareto analysis showed that the major cause for part rejection was an under size condition on the critical 84 mm diameter dimension. Per the standardized QC testing of each premix lot, all lots produced met the established specification for both absolute DC and difference from standard. However, it was acknowledged that either the standard testing was inappropriate or a new specification and /or a new standard lot were required. To produce immediate results, production of the premix was modified to produce greater sintered dimensional change. This was initially accomplished by substituting ~10% of the standard size copper (-150 micron) with fine copper (-15 micron). This proved successful but required lot-to-lot adjustments of 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 dimensional change.

Prior to any changes to the premix; the absolute dimensional change (DC) and difference from standard (DFS) of DC were monitored and recorded. The difference from standard (DFS) varied 0.06% before the change in copper type was applied, as seen in Figure 5. This value was well within the original specification jointly developed. As noted, analysis of the reject causes showed that the major cause was undersize on the critical 84 mm diameter. Thus, the change implemented was a transition to utilizing fine copper (-15 microns). In doing so, the DC DFS was reduced from a max of 0.06% to a max spread of 0.04%. How this initial change affected the production experience is shown as Figure 6. Prior to the start of this effort, non-conforming parts comprised approximately 4.5% of the total production. This high rate of non-conformity had a pronounced impact on productivity and, ultimately, cost of production. After the implementation both resetting the specification limits and replacing the copper addition as fine copper, the non-conformity rate was reduced to about 0.51%. It was also rationalized that the use of the fine copper enabled more complete chemical bonding of the copper alloy addition to the iron powder. As such, this promoted greater uniformity of the copper distribution with a corresponding improvement in flow rates of the powder. Total production experience showed that this premix resulted in considerable enhancements in total part processing and reduced final inspection costs.

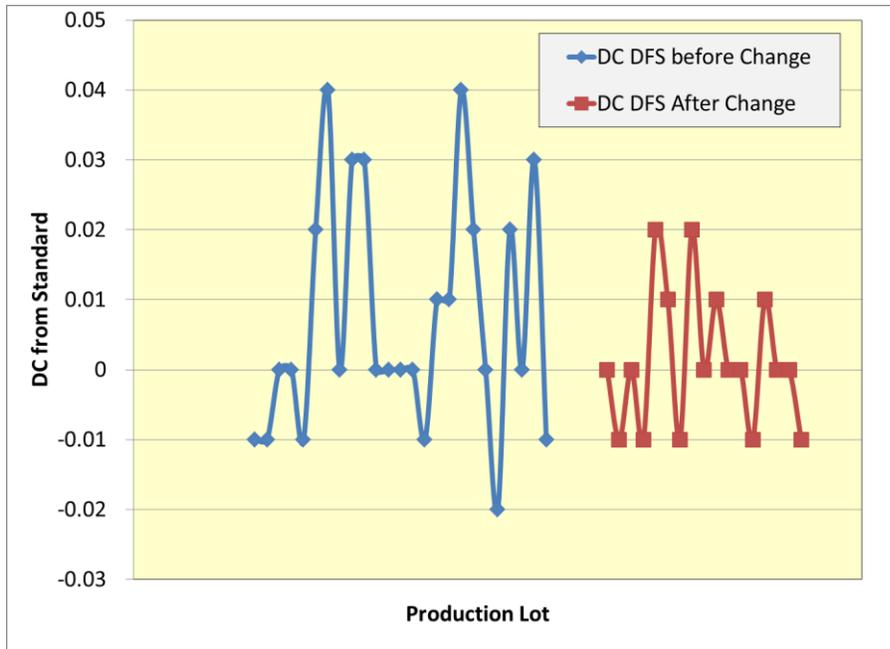


Figure 5: Dimensional control of FC-0208 premix used for VVT, using TRS bars compacted to a 7.0 g/cm³ green density.

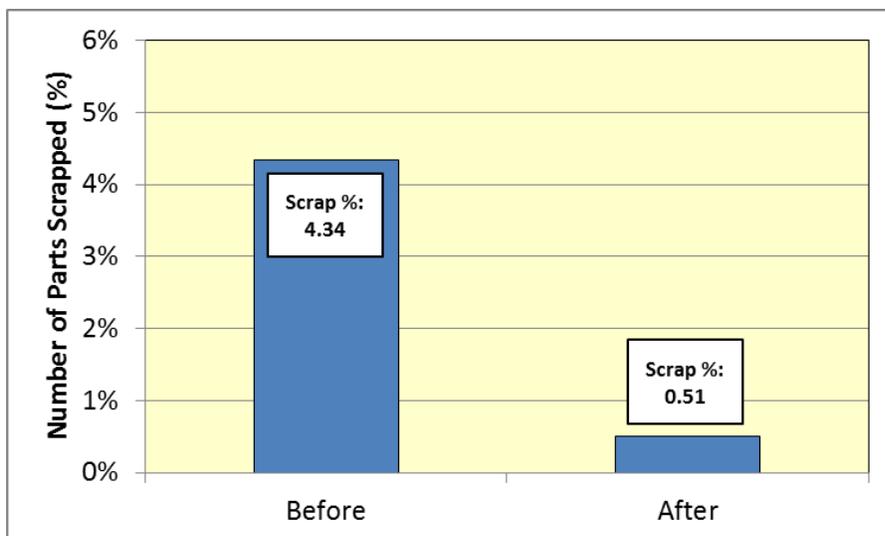


Figure 6 Reject rates before and after the various changes

Laboratory studies focused on the sintering response of coarse vs. fine copper additions to the FC-0208 premix. Shown in Figure 7 is the sintering response of two production lots, TRS bars were compacted to 6.6 g/cm³ and 7.0 g/cm³ green densities. These two

lots were specifically chosen because they represented material that showed a 0.08% difference in DC in both QC testing and in actual part production. The data presented in Figure 7 utilized 100% of the -150 micron copper powder. Nearly 100% of the dimensional change variation occurred after the copper melted. Unfortunately no differences in particle size analysis, powder morphology or particle surface oxides were found to explain this difference in behavior.

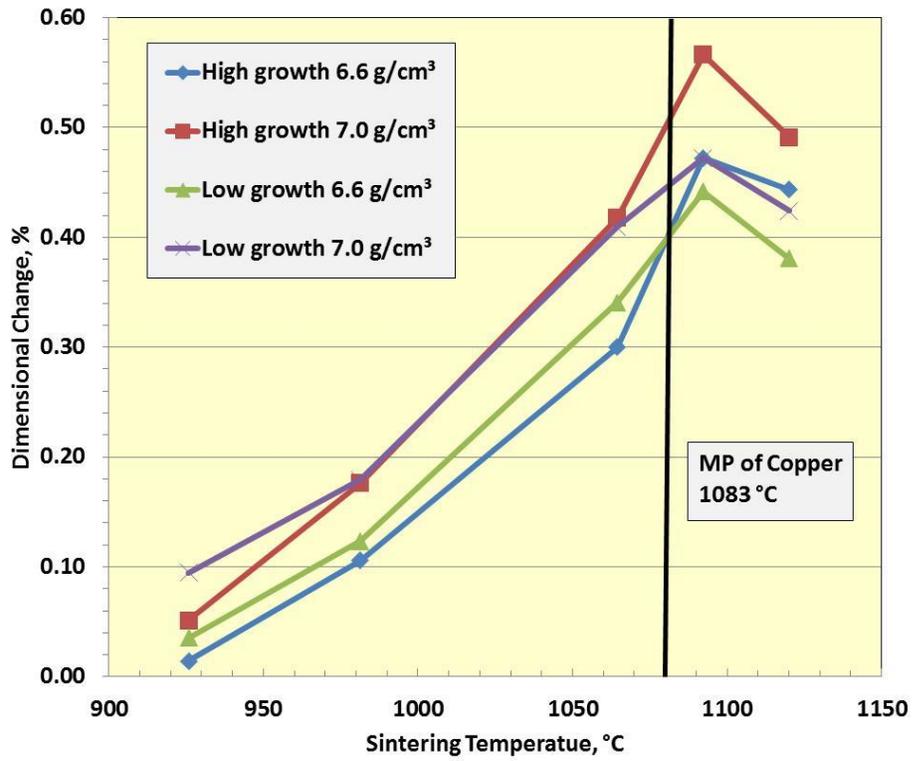


Figure 7: Dimensional change of two premixes with DC +/- 0.4 of the nominal value

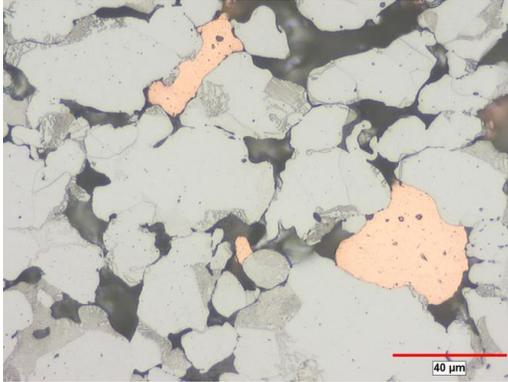


Figure 8a: Sintered at 845 °C

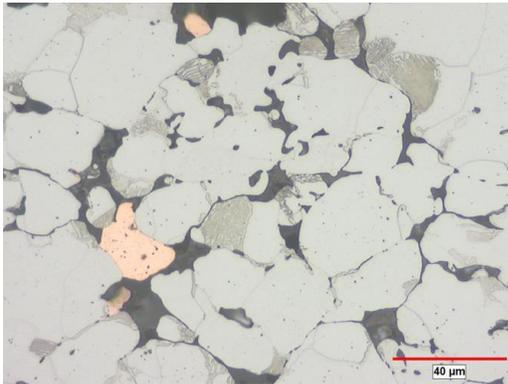


Figure 8b: Sintered at 870 °C

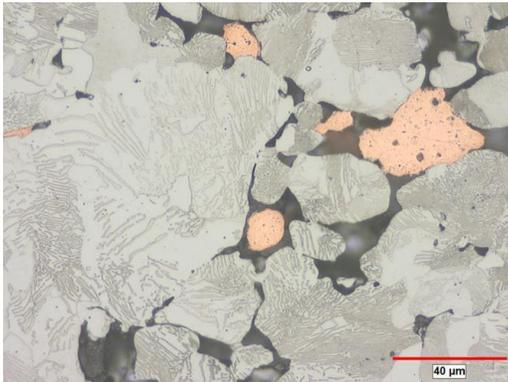


Figure 8c: Sintered at 980 °C

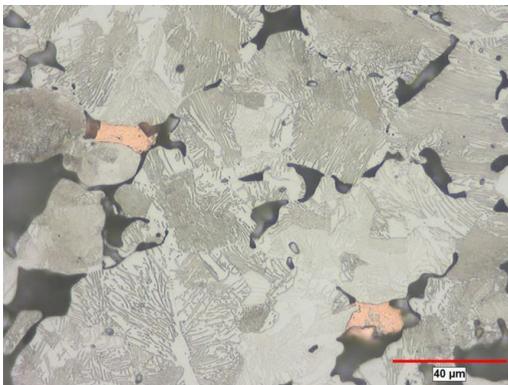


Figure 8d: Sintered at 1065 °C

Figure 8: Photomicrographs showing the carbon diffusion at temperatures ranging from 845 °C to 1065 °C.

A metallographic analysis of the conditions presented in Figure 8 showed that the onset of graphite diffusion is about 1550 °F (843 °C) and 100% of the graphite is in solution at approximately 1750 °F (954 °C), Figure 8c. Thus the dominant variable associated

with the copper growth is the melting and diffusion of the copper. Smaller particle size copper did not alter the graphite going into solution. Even with the fine copper addition, the copper particles are readily apparent at temperature up to 1065 °C. Murphy has reported that just below the melting point of the iron, the fine copper particles show significant solid state diffusion. [6] This would explain the higher growth associated with the smaller copper particle sizes.

As noted above, fine copper additions have the disadvantage of giving higher growth for an equivalent weight percentage addition. This experimental work suggested that a 1.85% fine copper addition gave the same growth as a 2.1% addition of the regular copper. This 1.85% addition is within the specification limits for an FC-0208 and a minor reduction in TRS strength was observed; however, the TRS strength met the nominal limits established in MPIF Std. 35 for FC-0208.

One final comment, as with all fine particle additions, additions of the fine copper are best added via high shear mixing techniques. This promotes greater copper uniformity throughout the premix and minimizes potential agglomerates of the fine particle sizes. Either metallurgical or chemical bonding is advantageous to prevent potential segregation of the fine particle additions.

Summary

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

1. It is extremely important to specify the correct standard and specification limits for any PM part. Choosing an inappropriate standard can lead to potentially high rates of non-conforming parts.
2. Careful design of the tooling is necessary to compensate for the differences in dimensional change resulting from varying part densities. This is particularly valid in copper steels.
3. Utilizing smaller particle size copper additions reduce or eliminate large pores resulting from copper melting. This has the potential advantage of improved mechanical properties.
4. Using a -15 micron copper particle size vs. a -150 micron particle size necessitates that lesser amounts of copper be used. The fine copper addition has a significant large number of copper-iron particle contacts; thus, promoting greater copper diffusion into the iron.
5. With careful control of the premixing and utilizing the proper standards, it is possible to maintain a + / -40 micron tolerance on an 84 micron diameter.
6. Significant reductions in non-conforming parts were realized with a corresponding reduction in inspection costs and improvements in part productivity.

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