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Improvement of Dimensional Stability of Sinter Hardening Powders under Production Conditions

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ABSTRACT

Sinter hardening of steel powders in furnaces equipped with fast cooling units represents a cost effective way to manufacture components with high strength and high apparent hardness without the need for secondary operations. However, due to microstructural transformations allowing achievement of high hardness after sintering, the dimensional control of components is of paramount importance. For this reason, Rio Tinto Metal Powders and Metaldyne, a powder producer and a part manufacturer, respectively, have worked together over the last few years in order to better understand the behavior during sintering of ATOMET 4601 and ATOMET 4701, two widely used sinter-hardening powders. Extensive studies were carried out under production conditions with the ultimate goal to improve the dimensional accuracy of components and to reduce as much as possible the cost related to quality issues resulting from dimensional variation.

Some of the key factors related to the materials and to the sintering process, as well as their impact on dimensional change, are discussed in this paper. In the first part, the effect of mix formulation and type of additives on dimensional change occurring during sintering is evaluated. In addition, the optimized mix formulations obtained at the end of the first part and their properties are summarized. In the second part, the effect of sintering conditions on dimensional consistency is presented. In particular, the improvement in dimensional consistency achieved through optimization of sintering conditions in mass production is discussed in detail. In conclusion, it was found that both materials and processing conditions must be optimized in order to maximize the benefits offered by the sinter hardening technology.

INTRODUCTION

In order to successfully meet higher requirements for new challenging applications at a competitive cost, high performance steel powder grades are sintered in furnaces equipped with fast cooling units to produce parts with high mechanical strength and hardness, thus avoiding post-sinter heat treatments. This is the case for ATOMET 4601 and 4701, two powders produced by RTMP, which are widely used in sinter-hardening applications.

Due to the high apparent hardness reached after sintering, a very tight control of final dimensions of the parts is required, since it is almost impossible to adjust the final size by secondary operations [1]. In order to achieve such a tight dimensional control on a day-to-day basis in mass production, several conditions must be met, such as: 1) the key powder characteristics affecting dimensional change must be very well controlled during manufacturing, 2) mix formulation and additive grades must be carefully selected, 3) each manufacturing step involved in the production of sintered parts must be tightly controlled and optimized. A very good understanding of the behaviour of sinter-hardened powders during the sintering cycle is essential to meet these conditions. In this regard, many studies were carried out during the last decade, mainly using dilatometry, to better understand and quantify the influence of various factors on the dimensional change of ATOMET 4601 and 4701 [2]. Dilatometry is a very powerful tool to study the sintering behaviour of materials, as proven by many researchers [1,3-10]. The results of these studies have led to the development of a relatively low cost grade of graphite, CarbQ, used in the production of pre-mixes with improved dimensional consistency. However, in the most recent years, the efforts have been mainly focused on the optimization of sintering conditions to further improve the dimensional robustness.

It is the objective of this paper to review the effect of graphite and copper on the dimensional change behaviour in the first section and discuss the dimensional performance of optimized materials under standard and optimized mass production conditions in the second section.

EXPERIMENTAL PROCEDURES

Table 1 gives the chemical composition for ATOMET 4601 and 4701, named powder 1 and 2 respectively. The difference in hardenability between the two powders is illustrated in Figure 1. As shown, powder 2 is more hardenable than powder 1.

Table 1. Chemical composition of ATOMET 4601 and 4701.

Grade	Ni, %	Mo,%	Mn,%	Cr,%	O,%
Powder 1 (ATOMET 4601)	**1.8**	**0.55**	**0.2**	**0.05**	**0.10**
Powder 2 (ATOMET 4701)	**0.9**	**1.0**	**0.5**	**0.5**	**0.20**

Lab-scale sintered properties were measured on standard TRS specimens pressed either at 6.8 or 6.9 g/cm³. Sintering was performed in a mesh belt furnace at around 1140°C for 25 min in a 90%/10% nitrogen/hydrogen atmosphere. The cooling rate achieved in the temperature range 650°C to 400°C was ~ 0.8°C/sec. The dimensional response was evaluated either with a Theta dilatometer model DILATRONIC 1600°C or Linseis dilatometer model L75. For all tests, the specimens were heated at a rate varying from 10 to 20°C/min to reach ~1120°C for 40 minutes, and then cooled at the maximum capacity of the dilatometers in an atmosphere consisting of pure Argon, Argon with 5% H_2 or 90%N_2/10% H_2. All samples were previously treated at 600°C in a nitrogen atmosphere to remove the lubricant.

The effect of sintering conditions in mass production was evaluated at Metaldyne in 61 cm wide mesh belt furnaces equipped with fast cooling units. The material selected was powder 2 admixed with 1.7% copper, 0.85% CarbQ, 0.5% MnS. Tests were carried out either on long rectangular bars (12.3 cm x 2 cm x 1.3 cm) or on sprockets with a diameter of ~ 138 mm pressed to a density of about 6.8 g/cm³ in mechanical presses. For each test, parts were placed in the middle of a production run or between dummy parts placed upstream and downstream to get stabilized sintering conditions. Sintering atmosphere was nitrogen with 5-10% hydrogen.

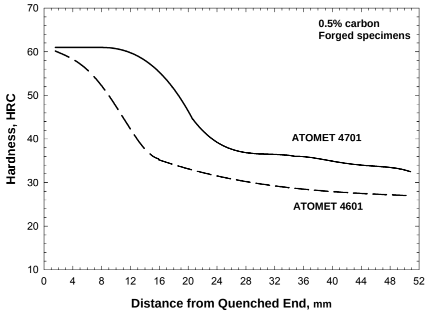


Figure 1. Jominy curves for ATOMET 4601 and 4701.

EFFECT OF POWDER FORMULATION ON DIMENSIONAL BEHAVIOR

Cu-graphite interactions during sintering

Copper and graphite additions are both used to achieve sufficiently high hardness after sintering. Figure 2 illustrates the variation of dimensional change as a function of sintered carbon content for 0, 1 and 2% copper for powders 1 and 2. It should be noted that a very similar trend is obtained when fast cooling and/or tempering are applied on parts. The dimensional response as a function of sintered carbon is highly affected by the copper content. Indeed, without copper, dimension of parts increases with combined carbon while trend is reversed when copper is added. At 2% copper, the dimensional change response is highly affected by the combined carbon content, especially above 0.5% for both powders, which corresponds to the formulations giving the highest hardenability. Dimensional change varied by about 0.01% for each increment of 0.01% in sintered carbon under these conditions. Dimensional change is much

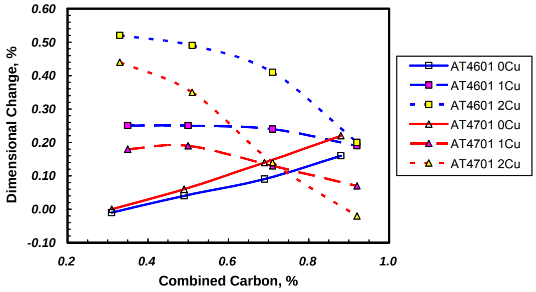


Figure 2. Dimensional change as a function of carbon and copper content.
Density: 6.9 g/cm³, cooling rate: 0.6°C/sec

more stable at 1% copper. However, hardness can be significantly lower and such an option is probably not possible if the cooling rate is relatively low because of part mass, furnace cooling capabilities and/or furnace loading. This is particularly true with powder 1, which shows lower hardenability. For this reason, powder 1 is normally admixed with around 2% copper and 0.7% graphite or more, in order to maximize hardness. On the other hand, reducing copper content down to 1% has very little effect on hardness for powder 2.

Figure 3 illustrates the dimensional change response of powder 1 with 2% copper and 0.9% graphite as measured by dilatometry. Five major transformation events can be observed during the heating/cooling cycle: (1) α→γ phase transformation, (2) carbon diffusion (from graphite), (3) copper melting and growth, (4) contraction at isotherm and (5) γ→α+carbides phase transformation. The variation in size observed outside these major areas corresponds to the thermal expansion/contraction of α or γ. It was determined that the two phenomena having the strongest effect on the final dimension were the carbon diffusion (2) and the copper melting (3) [1]. In particular, the growth associated with the copper melting, called copper growth, is a function of both the concentration of copper and carbon. Larger copper growth is obtained when copper content increases and carbon content decreases [1].

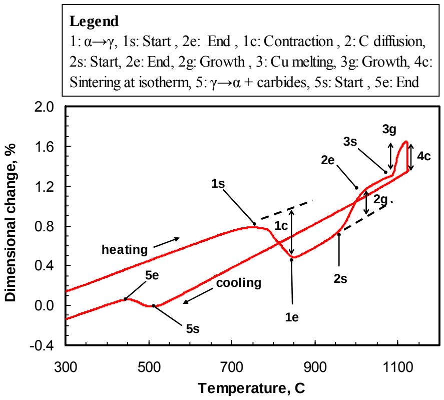


Figure 3. Typical dilatometry curve for powder 1 with 2%Cu and 0.9%C.

In addition to the level of graphite, it was also found that the copper growth was affected by the diffusion behavior of graphite, which is mainly a function of the type and size distribution of graphite and the nature of the steel base powder. This is well illustrated in Figure 4 for powder 1 with 2% copper and 1.0% graphite. The carbon diffusion starts and ends at a much lower temperature in the case of synthetic graphite. Even if the total growth associated with the carbon diffusion prior to the copper melting is basically the same for both types of graphite, the copper growth was strongly reduced with synthetic graphite. The lower copper growth obtained with synthetic graphite is likely related to a better iron diffusion and inter-particle necking prior to the copper melting, as shown in Figure 5. Indeed, the expansion rate obtained after the completion of graphite diffusion is lower than the thermal expansion rate of γ, clearly indicating that iron diffusion takes place. The expansion rate with synthetic graphite is clearly lower than that with natural graphite, indicating faster iron diffusion.

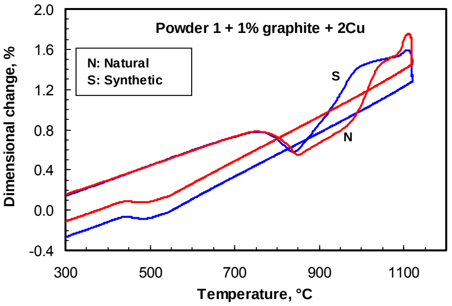


Figure 4. Effect of graphite type on carbon diffusion and copper growth.

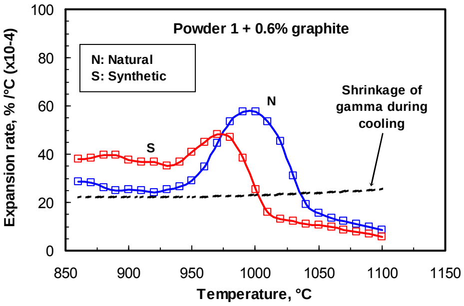


Figure 5. Expansion rate as a function of temperature for different grades of graphite.

The effect of carbon diffusion on the copper growth is illustrated in Figure 6 for powders 1 and 2 admixed with 2% copper and 1% graphite. As shown, a reduction of the temperature at which carbon starts to diffuse leads to a reduction and stabilization of the copper growth. It is quite obvious from that figure that the use of graphite with excellent carbon diffusion characteristics would result in a greater dimensional stability. It is also clear from that figure that the diffusion of graphite in powder 2 is a little bit more difficult. This is likely related to the higher level of oxygen for this powder grade.

Dimensional consistency of optimized production-scale mixes

In light of results of dilatometry studies, a new grade of graphite, CarbQ, with excellent diffusion behavior and lower cost compared to synthetic grades was introduced a few years ago. More details on the characteristics of this new graphite can be found in reference 11. Table 2 gives statistical information related to the dimensional change measured in a QC laboratory furnace for production-scale mixes of powders 1 and 2 containing natural graphite and CarbQ [2]. For both powders, the standard deviation measured and calculated is about 40% lower in the case of mixes with CarbQ.

Table 2. Dimensional change variability for pre-mixes with natural graphite and CarbQ. Mix: 2Cu, ~1.0 Graphite12].

Powder	Standard deviation measured	
	Natural	CarbQ
1 (AT4601)	0.029	0.018
2 (AT4701)	0.034	0.023

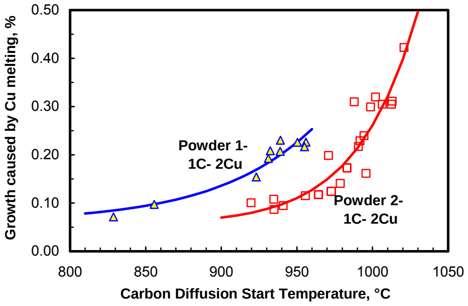
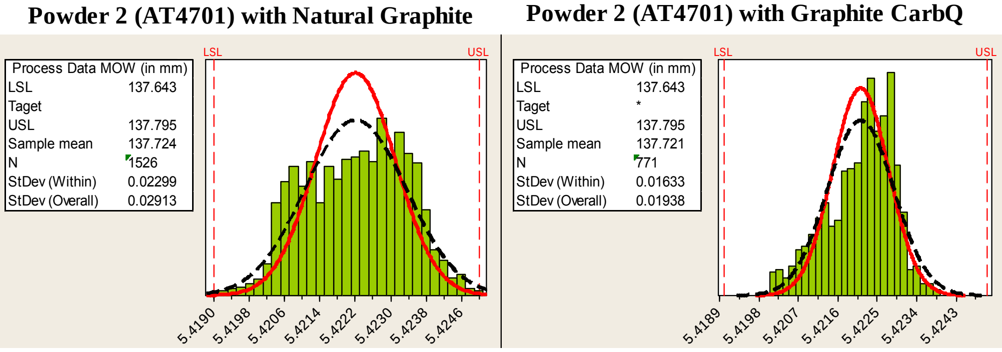


Figure 6. Effect of carbon diffusion start temperature on copper growth.

The benefit of CarbQ on the dimensional consistency of heavy parts processed in mass production under the same conditions, is illustrated in Figure 7, where the distribution of the outer diameter, obtained with the "measurement over wire" method (MOW), for powder 2 admixed with natural graphite and CarbQ is summarized. It is clearly shown that standard deviations are significantly reduced with CarbQ and, as a result, the PpK (Critical Process Capability) is significantly improved from 1.03 to 1.5.



Process Data MOW (in mm)	
LSL	137.643
Taget	
USL	137.795
Sample mean	137.724
N	1526
StDev (Within)	0.02299
StDev (Overall)	0.02913

Process Data MOW (in mm)	
LSL	137.643
Taget	*
USL	137.795
Sample mean	137.721
N	771
StDev (Within)	0.01633
StDev (Overall)	0.01938

Figure 7. Statistical variation of outer diameter (MOW) obtained during production of sprockets with a mix containing either natural graphite or CarbQ.
Mix: 2%Cu -0.9%C (MOW is given in inch in the distribution figures).

IMPROVEMENT OF SINTERING PERFORMANCE IN MASS PRODUCTION

Background

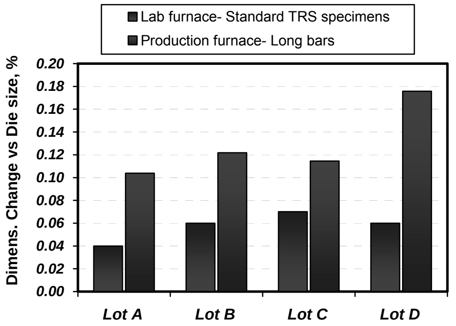


Figure 8. Dimensional change measured in lab and production-scale conditions (Powder 2).

Although the change in graphite grade resulted in a significant reduction of the part-to-part size variation as shown in figure 7, the sintering performance of sprockets produced with powder 2 was not yet optimal for two reasons. First, the correlation between the sintered size achieved in laboratory and the size achieved on production parts was not always good. This fact is well illustrated in Figure 8, which shows the dimensional response of different lots of powder 2 admixed with 1.7% copper, 0.85% graphite, and 0.5% MnS sintered in laboratory and production-scale furnaces. Although all lots gave very similar dimensional change on TRS specimens sintered in laboratory, lot D gave larger dimensional change than the others under mass production conditions. Even though the increase in growth was relatively small (< 0.07%), it is well known that it still has a significant effect on the quality and, as a result, on the level of rejected parts. Thus, it remains crucial to center as much as possible the size in the middle of the specification by adjusting sintering conditions.

Secondly, the material did not behave necessarily as expected when adjusting sintering conditions. Indeed, readjusting the size of a part is done mainly through sintering temperature and belt speed adjustments. For example, in order to increase the shrinkage of a part, higher sintering temperatures and/or lower belt speeds, which result in longer times at temperature, are used. Nevertheless, in some cases where oversize parts were obtained, it was observed that these changes did not result in smaller size, as shown in Figure 9.

There was thus a need to understand the reason for such a behavior and find ways to improve the dimensional consistency of sinter-hardened powders under mass production conditions.

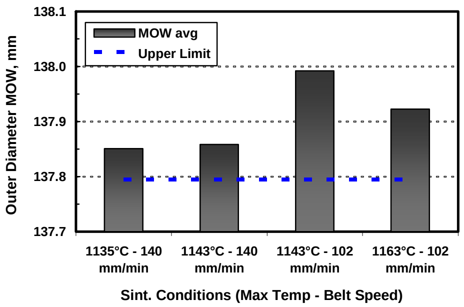


Figure 9. Effect of sintering conditions on size for sprockets produced with powder 2.

Effect of temperature profile

As a first step, dilatometry was performed in a Linseis dilatometer at a rate of 20°C/min on lots A and D from Figure 8 in order to verify if there was some difference in the behavior of these two materials. Dilatometry curves for these two powders are shown in Figure 10, where typical dilatometry curves obtained with natural and synthetic graphite and extracted from a previous study are included as reference. As shown, both powders behave in a very similar way, almost identical to that of the mix with synthetic graphite. In both cases, graphite diffusion started and ended at very similar temperatures, ~890 and 1050°C, respectively. Also, graphite diffusion ended prior to copper melting. As a result, very similar copper growth, between 0.10 and 0.13%, was observed. Finally, the shrinkage obtained at the sintering temperature was very similar for both powders. Therefore, the difference in size illustrated in Figure 8 could not be explained by a difference in material characteristics.

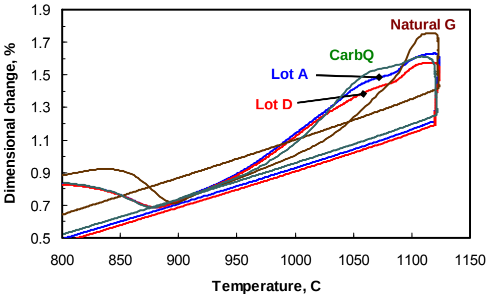


Figure 10. Dilatometry curves for Lot A and D (Powder 2 + 1.7Cu + 0.85C + 0.5MnS).

The fact that an increase in sintering temperature or a reduction of belt speed had almost no effect on sintered size, as illustrated in Figure 9, clearly suggests that a phenomenon occurs during sintering that counteracts the larger shrinkage that should be obtained from the increase in sintering temperature and/or time. It was shown in Figure 6 that copper growth, which is affected by graphite diffusion, can vary quite significantly, from 0.10 to ~ 0.40%. The copper growth is thus of the same order of magnitude as the shrinkage at maximum temperature. Graphite diffusion, like any other diffusion process, is time related. It was then decided to investigate the temperature profile. Temperature profiles were recorded with a thermocouple inserted in the core of a part positioned in the middle of the belt. Time between 875 and 1080°C ($T_{875-1080^\circ C}$) during heating was then calculated. $T_{875-1080^\circ C}$ corresponds to the temperature range where carbon diffusion may occur before copper melting. Figure 11 illustrates the $T_{875-1080^\circ C}$ for laboratory and production-scale furnaces and dilatometers at 20°C/min. As shown,$T_{875-1080^\circ C}$ was very low (only ~5.5 min for production-scale furnaces) compared to about 30 min for the laboratory furnace and 10 min for dilatometry tests at 20°C/min. The time required to complete the carbon diffusion from dilatometry tests was around 7 min. $T_{875-1080^\circ C}$ obtained for production furnaces was likely too short to ensure complete diffusion of graphite prior to copper melting and was likely responsible for the difference in size shown in Figure 8 and the lack of correlation.

In order to confirm the effect of time spent between 875 and 1080°C during heating on the dimensional change, sintering tests were run with 4 different lots of a mix with powder 2 (1.7%Cu-0.85%C-0.5%MnS), including lot D that gave larger growth. Sprockets were sintered with conventional furnace settings and modified furnace settings to increase the carbon diffusion time. Two tests were run with modified settings to validate the effect of time in the sintering zone. Temperature profiles were recorded to enable the measurement of $T_{875\text{-}1080°C}$ (carbon diffusion zone) and the time above 1095°C (sintering zone). The sintering conditions as well as the dimensional change measured for the outer diameter for these three tests are given in Figure 12, where the dimensional change measured in laboratory is also included. As shown earlier, a much larger difference in size between lots was obtained with conventional (0.05%) and laboratory (0.01%) sintering conditions. The use of modified settings resulted in a much more stable dimensional change, with the difference between lots being reduced by almost 80%. In particular, it was observed that the dimensional change of lot D, that showed the largest growth amongst the lots tested with conventional settings, shifted by about -0.03 to -0.04% with modified settings, even though the time above 1095°C and the maximum temperature were lower. This result confirms the importance of graphite diffusion and copper growth on the final sintered size of components. On the other hand, the increase in time above 1095°C with MOD-B conditions resulted in a shift in dimensional change for the 4 lots by about -0.01% versus MOD-A conditions. This result was in line with expectations.

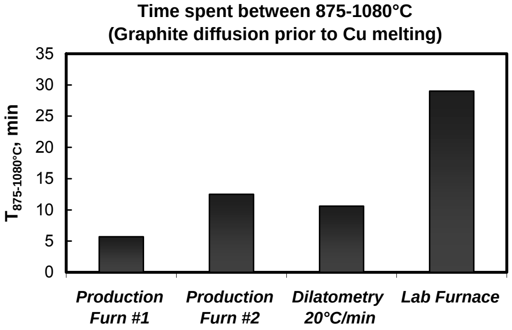
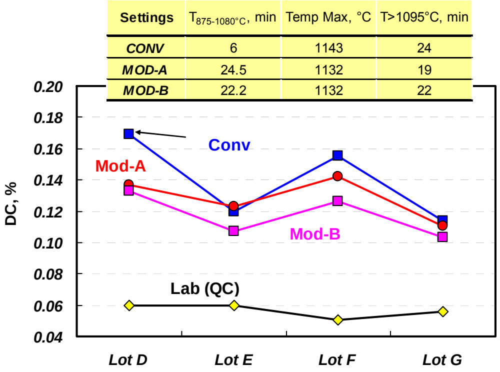


Figure 11. Time between 875 and 1080°C for different sintering furnaces.



Settings	$T_{875\text{-}1080°C}$, min	Temp Max, °C	T>1095°C, min
CONV	6	1143	24
MOD-A	24.5	1132	19
MOD-B	22.2	1132	22

Figure 12. Effect of sintering conditions on dimensional change of different lots of powder 2. Mix: powder 2 – 1.7Cu-0.85C-0.5MnS

Dimensional consistency of optimized sintering conditions

The effect of graphite change and the implementation of a new sintering profile on the dimensional change consistency of components produced with two different pre-mixes with powder 2 are summarized in Figure 13. For the mix with 2% copper, changing the graphite from a natural grade to CarbQ gave a significant reduction of standard deviation, which resulted in an almost 55% improvement in the critical process capability (PpK). The optimized sintering conditions contributed

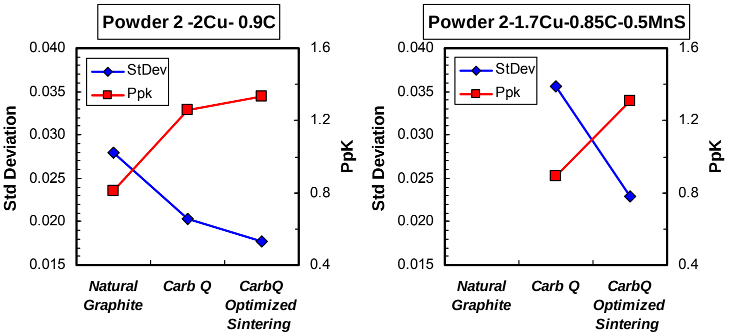


Figure 13. Statistical variation of the outer diameter (MOW) for sprockets produced with two different mixes with powder 2.

to a further improvement of 10% of the process capability. For the mix with 1.7% copper and 0.5% MnS, CarbQ was selected from the beginning. However, the standard deviation was quite high. The use of optimized sintering conditions together with CarbQ resulted in a significant reduction of standard deviation and an increase in PpK of about 50% compared to conventional sintering conditions.

CONCLUSION

The effect of graphite and copper additions as well as the effect of the sintering conditions on the dimensional behavior and consistency of ATOMET 4601 and 4701, two powders used for production of sinter-hardenable components, were studied and discussed in this paper. The major conclusions that can be drawn are:

- The graphite diffusion characteristics prior to copper melting and copper growth have a great influence on the final dimensions of a sintered component and greatly affect the part-to-part consistency.
- The copper growth phenomenon in ATOMET 4601 and 4701 with 2% copper and 0.9% graphite is of the same order of magnitude as the shrinkage obtained at maximum temperature.
- CarbQ, a graphite grade with excellent diffusion characteristics was introduced in pre-mixes to reduce the dimensional variability. Introduction of this graphite grade resulted in a reduction of the lot-to-lot standard deviation by 40% and the part-to-part standard deviation by 55% under mass production conditions.
- From dilatometry tests, it was observed that CarbQ diffusion temperature range in ATOMET 4701 is between 875 and 1025°C under excellent reduction conditions. This process takes about 7 minutes to be completed.
- The time available for graphite diffusion in steel particles prior to the copper melting point is also crucial during sintering of components in mass production. It must be sufficiently high to ensure a complete diffusion of carbon from graphite, a relatively low and constant copper growth and lower dimensional variation.
- Modified sintering conditions**,** allowing achievement of a much longer time between 875 and 1080°C and similar time at maximum temperature compared to conventional sintering conditions**,** were tested positively. Implementation of such conditions for production of heavy components resulted in a significant reduction of part-to-part and lot-to-lot size variation.

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