

Dimensional Change Characteristics and Surface Quality of Fully Dense Iron Base Infiltrated Parts

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ABSTRACT

The present study examines the factors effecting the dimensional change characteristics and surface quality of iron base infiltrated parts. The dimensional change characteristics include part uniformity as well as part to part consistency. The surface quality relates to the disposition and effects of infiltrant residues on the structural integrity and appearance of the infiltrated surfaces.

It is shown that part uniformity and part to part consistency are dependent on essentially the same factors that determine dimensional variability in ordinary P/M but are inherently more sensitive to these factors due to the liquid phase sintering effects that accompany the process. Relative to infiltrated surface quality, it is shown that the condition of the infiltrated surface is primarily dependent on the composition of the infiltrant residue and hence, penultimately on the composition of the infiltrant. Thus, reasonably defect-free infiltrated surfaces are obtainable by control of the infiltrant composition.

INTRODUCTION

The results of continuing research to develop iron base infiltration as a viable parts making process have been reported in three earlier papers (1, 2, 3). The first two of these detailed the fundamentals of the process and the third indicated its potential in terms of mechanical properties. To adequately define the conditions needed to implement the process as a practical matter, it remains to delineate the factors effecting the dimensional change characteristics and the surface quality of the resulting parts.

Dimensional Change Issues

The dimensional change characteristics of the infiltrated compositions according to the present technology are generally more sensitive to processing than in regular P/M. Potentially, this sensitivity could prove to be a significant advantage of the technology. However, as set out below, it has the possibility to be problematic as well.

In part, the indicated processing sensitivity is a consequence of the fact that the carbon contents of the present compositions are essentially fixed at values that are typically of the order of 2 % or higher. Thus, depending on processing, the average dimensional change of the resulting parts has the potential to be

high as well. For example, values of the order of 1% or higher are easily achieved simply by using a relatively low infiltration temperature and a sufficiently high infiltrant weight to preclude shrinkage by sintering after infiltration. High dimensional change values, however, are generally held to be undesirable as they are typically accompanied by high dimensional variances in the resulting parts (4). In addition, high values are often anisotropic and hence, have the potential to complicate both the design and quality control phases of the parts making process.

At the same time, the present compositions are also eminently liquid phase sinterable. Thus, the alternative possibility exists to reduce the average dimensional change to relatively lower values by designing the infiltration process to effect large sintering shrinkages. In general, this involves increasing the process temperature and decreasing the infiltrant weight to provide sufficient residual porosity to effect the desired shrinkage by liquid phase sintering after infiltration. The potential drawback in this case, however, is that large sintering shrinkages may cause within part non-uniformities that are at least as undesirable, if not more so, than the part to part variations that they are designed to preclude (5).

In consideration of these possibilities, it will be evident that studies were needed to decide what general processing to recommend to parts makers. Thus, the present studies were conducted with this aim. The specific objectives in this regard were to determine the effects on both part uniformity and part to part consistency of two different processes: one designed to produce an high average dimensional change; and the other, to produce a relatively much lower one.

Infiltrated Surface Appearance

The infiltrant composition according to the present technology is a standardized Fe-C-Si alloy that is designed to melt by a eutectic reaction that occurs at approximately 1154 °C (2109 °F) . It is purposely made slightly hyper-eutectic in carbon content to provide for the inevitable graphite dusting and oxidation losses that typically occur during processing in advance of infiltration. The base compact composition is also a standardized Fe-C-Si alloy. In contrast to the infiltrant, it is made precisely to the eutectic solidus composition of the alloy. However, in this case, dusting and oxidation losses in advance of infiltration plus incomplete homogenization of the admixed silicon units it contains cause it to be slightly hypo-solidus in carbon content at the instant of infiltration. Consequently, provided that its surface is thermally equilibrated with the infiltrant at the onset of infiltration, it is not subject to appreciable erosion by the eutectic liquid during infiltration. Nevertheless, as explained below, the infiltrated surface is susceptible to erosion after infiltration.

As it turns out, the infiltrant does not melt completely during the infiltration process because it contains a small percentage of non-fusible components. These primarily include refractory oxides, sulfides and graphite. The oxides and sulfides derive mainly from the impurities of the base iron and graphite constituents of the infiltrant. The graphite is a residual of the infiltrant's initially hyper-eutectic carbon content. As the fusible constituents melt and infiltrate the base compact, the non-fusible ones separate out and deposit on the infiltrated surface as a residual matte of loosely adherent particles. The matte either initially forms or later aggregates into a miniature replica of the original infiltrant slug. As the temperature of the system rises to the set point temperature which is necessarily above the eutectic temperature, the carbon units in the matte become increasingly reactive and can, in accordance with the phase relations of an eutectic alloy, form additional liquid. However, since the metallic component of this liquid can now only come from the infiltrated surface, its formation will generally produce a surface defect.

In fact, studies of this have shown that the indicated secondary reaction of the surface of the base compact with the graphite in the infiltrant residue may produce many different types of defects. Some of the defects that have been observed represent little more than a cosmetic nuisance but others are decidedly

more problematic as they clearly threaten the structural integrity of the part. In the general case, the number and severity of the defects varies directly as the carbon content of the residue. Thus, the obvious implication with regard to mitigating the problem as a general matter is to reduce the carbon content of the residue to a suitably low level. Two strategies exist to do this: one is to effect the indicated reduction via the carbon potential of the sintering atmosphere; and, the other is to do it independently of the atmosphere by increasing the refractory oxide content of the infiltrant.

Limited studies to date have shown that each of these strategies is helpful. However, it still remains to determine the best infiltrant composition in terms of oxide type and content as well as the most effective processing. In the present study, the effects of minor changes, including increased oxide contents, to an existing infiltrant composition were examined.

EXPERIMENTAL PROCEDURE

Minimum Specimen Number

General experience with iron base infiltration indicated that to obtain a valid assessment of the effects of processing on part dimensions and dimensional change, it would be necessary to employ a procedure that included a large enough number of specimens to provide statistically significant indications of the parameters of interest as well as to demonstrate the existence of stable operating conditions during the course of the trial. The statistical parameters of primary interest were the average and the standard deviation. Of the two, the accuracy of the standard deviation is by far the more dependent on specimen number than the average and it was decided to select the specimen number accordingly. At the 95 % confidence level, the number of specimens required to estimate the standard deviation to within 20 % of its true value can be shown to be 47 (6). Although the 20 % accuracy figure is not high, it was considered adequate for present purposes. Moreover, higher accuracies were associated with exponentially higher specimen numbers. For example, at 10 % accuracy, the required specimen number was upwards of 185. In contrast, the value of 47 was both experimentally manageable and otherwise appeared reasonable in terms of demonstrating operational stability. Consequently, it was incorporated into the experimental design as the minimum specimen number.

Base Compact Geometry and Composition

It was also known from experience that the infiltrated compositions according to the present technology were dimensionally sensitive to green density variations even in the absence of significant sintering shrinkage. Consequently, in view of the introductory nature of the study, it was considered essential to design the procedure around a relatively simple part geometry that could be made with a reasonably uniform density distribution. The geometry that was eventually selected was the cylindrical one. It offered a high probability of radial uniformity with respect to density in the plane of pressing as well as the potential to compact parts with a reasonably centralized neutral axis of density in the direction of pressing. To maximize the metric accuracy of the results, it was decided to base the study on parts made from the largest diameter tools that were available in the laboratory. This diameter was just a little under 4.5 centimeters (~1.75 inches). The preferred base compact density according to the present technology is 6.7 g/cm³. The earlier mentioned studies had shown that this density is optimal both in terms of maximizing the infiltration rate and minimizing the infiltration weight to full density. The fill of the tools at this density, however, limited the aim height of the parts to just under 1.4 centimeters (~ 0.55 inches). This height was not optimal in that substantially higher heights were known to be possible and would have presumably provided a better indication of the true potential of the technology. Nevertheless, it had the advantage that it was more likely to result in a reasonably uniform density distribution in the direction of pressing than a higher height (7).

The composition of the base compact mix used in the study was 1.91 % C, 0.75 % Si, 0.45% Acrawax C, 0.1 % zinc stearate, and the balance Ancorsteel 1000B. The carbon was added in the form of Asbury grade 3032 HS graphite. The silicon was added in the form of a proprietary water atomized iron base pre-alloy containing 20 % Si. The Acrawax C was a product of the Lonza Division of IMS Company, Chagrin Falls, Ohio. The zinc stearate was a product of Baer Locher, LLC, Cincinnati, Ohio. A single mix weighing about 225 kgs (500 lbs.) was made and binder treated in accordance with the ANCORBOND[®] process (8).

Infiltrant Geometry and Composition

The infiltrant slugs for the study could have been made with the same tools as used for the base compacts. However, it was decided instead to make them with slightly smaller tools having a diameter of about 3.7 centimeters (1.45 inches). There were three reasons. First, the early studies of the process showed the existence of a novel dimensional non-uniformity (called the ‘distortion effect’ in earlier papers). The effect was characterized by density gradients in the through thickness direction of the infiltrated compact that were manifest as differences in its lateral dimensions to a depth of a few millimeters below the infiltrated surface. Later (un-reported) work showed that the magnitude of the effect decreased with increase in the cross-sectional area of the infiltrated surface and in any case, could be completely prevented by using an infiltrant slug of a slightly smaller cross-section than that of the infiltrated surface. The second reason was that a smaller infiltrant slug leaves a smaller infiltrant residue and thus exposes less of the infiltrated surface to the possibility of surface defects. The third reason was that the tool set in this case was designed for manual usage which in terms of the experimental situation was more convenient than the typical automated press set. For example, the manual set permitted the use of small bench size infiltrant mixes which at weights of 1 or 2 kgs (2 or 4 lbs) were easier, faster and more economic to make than the average pilot plant mix at 225 kgs (500 lbs).

The composition of the infiltrant mix used in the study was 4.4 % C, 0.2 % Si, 0.05% refractory oxide, and the balance Ancorsteel 1000B. The sources of the carbon and silicon admix additions were the same as used in the base compact composition. The refractory oxide was added in the form of a metallic soap which was known to calcine to the oxide during heating to the process temperature. Two such mixes were made weighing about 1.5 kgs (~ 3 lbs.) each. Here again, both were submitted to binder treatment processing after mixing.

As indicated at the outset, this infiltrant composition was experimental in that it was slightly different from the one that was previously in use to this point. Heretofore, the infiltrant was made with a small zinc stearate addition and to a slightly lower oxide content (i.e. 0.035 %). Early studies in the development of the technology had shown that a small metallic soap addition was helpful in homogenizing the mix and more particularly, in preventing graphite agglomerates. The indicated zinc stearate addition served this purpose. However, these studies pre-dated the findings that led to the use of the oxide and when it was later determined that the oxide was as effective to improve the surface appearance when added as a metallic soap as otherwise, it suggested the possibility to eliminate the stearate addition. Thus, the present composition was basically a test of this idea. Otherwise, the slight increase in the oxide content of the composition relative to the earlier one was essentially to consolidate its known effects in improving the infiltrated surface appearance.

Compaction Processing

Base compacts of the indicated geometry and density were pressed using a 200 ton Dorst production press. The press was set up so that the density split occurred approximately at the mid-height of the parts. The required compaction pressure was not measured but based on the compressibility of the mix was no greater than 485 MPa (35 tsi). A series of 350 parts were pressed at a rate of 10 parts per minute. The first 50 were set aside to be used in various preliminary studies and as dummy specimens to load the sintering

trays in advance of the trial specimens. Otherwise, the trial specimens per se were selected at random from the balance of the series.

Infiltrant slugs according to the technology are normally compacted to a pressure rather than a density. In the present case, slugs of the indicated geometry were compacted using a 60 ton Tinius Olsen press at about 485 MPa (35 tsi). As set out below, the infiltrant weight differed from trial to trial in accordance with the particular aims of the trial.

Infiltration Processing

In all, three infiltration trials were conducted: two to determine the dimensional change characteristics under different processing conditions; and, one to examine the effects of minor compositional and processing changes on the infiltrated surface appearance. The details were as follows.

The process conditions that were common to all three trials included the furnace, the furnace belt speed, the atmosphere flow rate, and the loading of the sintering trays. The furnace was a modified Abbott design equipped for both high temperature and sinter hardening operations. It was originally designed with a lubricant burn-off zone (LBO) and four high temperature zones but due to space limitations in the laboratory, was installed with only two hot zones. The belt speed in the trials was 5 centimeters per minute (2 ipm). With both hot zones set to the same temperature, the corresponding time at temperature was about 10 minutes per zone or 20 minutes total. The atmosphere flow rate was 330 liters per minute (700 cfh). The sintering trays {measuring about 25 cm (10 ins.) in length and made of alumina} were loaded with three specimens apiece. This loading was calculated to carry sufficient heat into the cooling zone to just heat the atmosphere to the process temperature. In addition, to stabilize the hot zone conditions in each of the three trials, seven trays of dummy specimens were processed ahead of the trial specimens and two such trays were processed behind them.

Trial 1 - The aim of the first trial was to determine the dimensional change characteristics corresponding to processing at the lowest practical temperature and the highest infiltrant weight. As mentioned, the eutectic temperature of the infiltrant composition was estimated to be about 1154 °C (2110 °F). This suggested a minimum process temperature of 1164 or 1165 °C (2125 or 2130 °F). Thus, both hot zones, in this case, were set to 1165 °C (2130 °F). Earlier studies had indicated that the infiltrant weight to full density of a base compact at a green density of 6.7 g/cm³ is minimally 15.7 % of the base compact weight. Thus, to allow for normal variations in pressing, the infiltrant weight was set slightly higher at 16.5 % of the calculated base compact weight.

Otherwise, the composition of the atmosphere in the trial was 75 % N₂ and 25% H₂ by volume. To protect the carbon units, especially of the infiltrant composition, methane was added to the atmosphere at the rate of ~ 0.1% by volume. Also, in the interests of consistency, the specimen number was increased to 48 (an integer multiple of 3) so that all of the test trays of the trial would be similarly loaded.

Trial 2 - The aim of this trial was to manipulate the process conditions to produce a significantly lower dimensional change, preferably one near zero or slightly higher, up to a maximum of about 0.25 %.. Based on experience with the present compositions, the plan was to increase the process temperature to about 1190 °C (2175 °F) and to decrease the infiltrant weight to provide enough residual porosity to just offset the dimensional change indicated in the first trial by liquid phase sintering after infiltration. As it turned out, the indicated decrease in the infiltrant weight was to 12.5 % of the base compact weight.

Experience had shown it to be prudent to approach the eutectic temperature gradually so as to minimize the temperature differences between the infiltrant and the base compact or else risk premature infiltration with serious adverse consequences to the outcome of the process. Thus, the first hot zone of the furnace in

this trial was again set to 1165 °C (2130 °F) while the second hot zone was set slightly above the aforementioned 1190 aim to 1195 °C (~2180 °F). In addition, to further increase the chances that the specimens would attain the aim temperature in the second hot zone, the N₂ and H₂ contents of the furnace atmosphere were reversed relative to those of the first trial. In other words, the composition of the atmosphere in this case was 75 % H₂ and 25 % N₂ by volume. This composition had about the same heat content as earlier but upwards of 2.5 times the thermal conductivity (9). Also, based on the general appearance of the infiltrated surfaces of the first trial, it was decided to eliminate the methane addition in this trial. In other respects, the specimen number, loading of the test specimen trays and the number and positions of dummy specimen trays were identical to those of the first trial.

Trial 3 - The aim of this trial was to provide additional information on the effects of the infiltrant composition and process conditions on both the infiltrant behavior and the infiltrated surface appearance. Basically, the effects of three modifications of the earlier composition were compared with those of the earlier composition. The indicated modifications of the composition were cumulative. They included: 1) returning the zinc stearate addition (i.e. 0.1 %) that had originally been excluded from the composition; 2) increasing the refractory oxide content from 0.05 % to 0.075 %; and, 3) again increasing the refractory oxide content to 0.10 %. As previously, the oxide additions were made in the form of a metallic soap.

In contrast with the earlier trials, only eight specimens (i.e. two per infiltrant composition) were processed in the trial. Otherwise, the process conditions in this case were generally similar to those of the first trial although there were three minor differences as follows: 1) the infiltrant weight was 15.7 % of the base compact weight, slightly less than the earlier 16.5 %; 2) the first hot zone temperature was set to 1157 °C (2115 °F), slightly lower than the earlier 1165 °C (2130 °F); and, 2) the methane addition to the furnace atmosphere was reduced to 0.05 %, again, down from the earlier 0.1 %.

Measurements and Calculation of Dimensional Properties

All measurements were made with micrometers and/or calipers that were calibrated in inches and were accurate to within 0.0001 inch (0.025 cm). The green diameters and heights of the base compact specimens were based on the average of two measurements per specimen. The two diametric measurements were at mid-height and at right angles to each other. The two height measurements were at the approximate center and at a randomly selected point near the outside edge of the specimen. The green expansions of the specimens versus the diameter of the die were calculated in the usual manner. The dimensions of the infiltrant slugs were not determined.

The infiltrated diameters of the specimens were measured in four places as follows: two each across the top and bottom surfaces in the parallel and transverse directions to their direction of travel through the furnace. The four values were then combined in different ways to assess three different dimensional characteristics including the dimensional change versus green, the concentricity and the top to bottom uniformity. The diametric value used to calculate the dimensional change and metric density properties was the average of the four values. The parameter used to indicate the concentricity was the absolute value of the difference between the averages of the two parallel and two transverse measurements. Similarly, the parameter used to indicate the top to bottom uniformity was the absolute value of the difference between the averages of the two top and two bottom measurements.

The infiltrated heights of the specimens were based on two measurements in the first trial and three in each of the succeeding trials. In all cases, the measurements were widely spaced with respect to each other and were usually made just outside the periphery of the infiltrant residue which was typically about 2 cm (0.75 ins.) in diameter and centrally located. In this case, the resulting values were simply averaged and used in the usual manner to calculate the dimensional change and metric density properties.

RESULTS AND DISCUSSION

Each of the three trials in the study provided valuable information on the effects of processing and composition on both the dimensional change characteristics and surface appearance of the specimens. However, the first two trials were specifically designed to examine the effects of processing on the dimensional change characteristics while the third trial had the aim to determine the effects of infiltrant composition on surface appearance. Consequently, as a practical matter, its desirable to divide the discussion of the results accordingly.

Dimensional Change Characteristics

The average green expansion of the base compact specimens versus the die was 0.076 %. The specimen to specimen differences in the green diameter values that contributed to this result were in all cases beyond the accuracy of the calibration of the micrometer that was used to measure them. Thus, the corresponding standard deviation value was negligibly small.

Trial 1 Results

The effects on the metric density and the three dimensional properties, as mentioned, of infiltrating the base compacts at a relatively low process temperature with a high infiltrant weight are shown below in Table 1. The statistics in the table include the average, the standard deviation, the maximum and minimum values of the sample population, their difference, and the quotient of the difference divided by the standard deviation. As will be recalled, the sample size in the trial was 48. To assess operational stability, the table also includes a listing of the averages and standard deviation estimates of four subsets of 12 specimens each that reflect the order of processing during the trial.

Table 1 - Results of Infiltrating at 1165 °C (2130 °F) With an Infiltrant Weight of 16.5% of the Base Compact Weight

Statistical Property	Metric Density	Diametric Dimensional Change	Absolute Δ Between Parallel & Transverse Diameters		Absolute Δ Between Top & Bottom Diameters	
	g/cm ³	% vs green	cm	(inches)	cm	(inches)
Average	7.40	1.29	0.0043	(0.0017)	0.0031	(0.0012)
Std. Dev.	0.018	0.051	0.0033	(0.0013)	0.0028	(0.0011)
Max.	7.44	1.39	0.0125	(0.0049)	0.0107	(0.0042)
Min.	7.37	1.19	0.0000	(0.0000)	0.0000	(0.0000)
Δ Max/Min	0.07	0.21	0.0125	(0.0049)	0.0107	(0.0042)
Δ/s	3.9	4.1	3.6		3.9	
<i>Subset Average and Standard Deviation Values</i>						
<u>Set 1</u> Nos 1 to 12	7.40 0.017	1.27 0.050	0.0038 0.0064	(0.0015) (0.0025)	0.0036 0.0048	(0.0014) (0.0019)
<u>Set 2</u> Nos 13 to 24	7.40 0.014	1.27 0.046	0.0043 0.0076	(0.0017) (0.0030)	0.0036 0.0038	(0.0014) (0.0015)
<u>Set 3</u> Nos 25 to 36	7.40 0.021	1.32 0.046	0.0048 0.0066	(0.0019) (0.0026)	0.0028 0.0020	(0.0011) (0.0008)
<u>Set 4</u> Nos 37 to 48	7.40 0.020	1.30 0.047	0.0046 0.0074	(0.0018) (0.0029)	0.0023 0.0023	(0.0009) (0.0009)

The estimated pore free density in this case is 7.54 g/cm^3 . In comparison, the average of 7.40 g/cm^3 that the above data show for the trial is about 98 % of this value. The normally expected density in this case was higher although not as high as the pore free value. For example, experience with these and similar process conditions has often shown values of up to 7.47 g/cm^3 but seldom higher than this. The 7.47 value is about 99 % of the pore free density and apparently indicates that only about 99 % of the porosity of the base compacts is readily accessible or quick to infiltrate under the indicated conditions. Thus, while the average density of the trial was not expected to be as high as the pore free value, it will be evident that the presently observed value of 7.40 g/cm^3 was still lower than what was known to be possible and hence, indicated the likelihood of a problem.

The simplest possibility in this regard was measurement error. For example, the metric density has generally not been found to be a very precise measure of the density of infiltrated specimens. More often than not, it underestimates their values and sometimes by a large margin (e.g. by as much as 0.05 g/cm^3 or more). However, as it turned out, this was only a part of the explanation in this case. Accordingly, immersion density determinations on a few randomly selected specimens confirmed the suspicion of higher values but the average difference was only about 0.02 g/cm^3 .

A review of the other findings of the trial suggested another possibility. Normally, the infiltrant residue weight averages less than 0.75 % of the infiltrant start weight. However, in this case, the residue weight was close to 6 % of the starting weight. The implication was that either a significant percentage of the infiltrant did not melt during the process or that it melted but did not infiltrate the compacts. In either case, the finding indicated an infiltrant problem. As will be seen, the results of the later trials both confirmed this indication and indicated that the cause was related to the refractory oxide addition

The balance of the density statistics in the table showed that the density variations were fairly modest. For example, the standard deviation was 0.018 g/cm^3 and the total range of the variations in the data as indicated by the $\Delta \text{ Max / Min}$ value was a little less than 1.0 % of the average or as alternatively indicated by the Δ/s parameter, a little less than four standard deviations. In addition, the similarity in the statistical values shown by the four subsets of the density data appeared to make a strong case for the idea of operational stability during the course of the trial.

The average diametric dimensional change from green shown in the table is 1.29 %. When combined with the earlier mentioned green expansion value, the corresponding average change from the die was 1.37 %. As expected, both values were high by ordinary P/M standards. However, the associated standard deviation value, at 0.051 %, was surprisingly low. More particularly, in terms of the average infiltrated diameter of the specimens {i.e. $\sim 4.5 \text{ cm}$ (1.77 ins.)}, it indicated a deviation of less than 0.0025 cm (0.0010 ins.) which at four deviations translated to a predicted range of 0.0100 cm (0.0039 ins) or, in effect, to a maximum diametric variation of $\pm 0.005 \text{ cm}$ ($\pm 0.002 \text{ ins}$). In actual fact, the infiltrated diameter data of the trial produced a standard deviation value of 0.0023 cm (0.0009 ins), a $\Delta \text{ Max / Min}$ range value of 0.0091 cm (0.0036 ins) and an diametric variation of $\pm 0.0045 \text{ cm}$ ($\pm 0.0018 \text{ ins}$). Otherwise, the values of the four subsets of the dimensional change data showed a little more variation than the density data but still indicated that the process had been reasonably stable through the course of the trial.

The average of the differences in the parallel and transverse diameters shown in the table is 0.0043 cm (0.0017 ins.). Although this parameter is admittedly not a quantitative indicator of concentricity, it nevertheless served to show that the specimens in this case were generally not concentric. Given the presence of the liquid phase and the known sensitivity of the infiltrated composition to liquid phase sintering, this was the expected result. Nevertheless, as a general matter, the magnitude of the value appeared to be reasonably moderate. However, this was not true of the standard deviation value which at

0.0033 cm (0.0013 ins.) was relatively large in comparison with it. As confirmed by the corresponding Max value {e.g. 0.0125 cm (0.0049 ins)}, the implication was that some of the individual values in the data were substantially (i.e. 2 to 3 times) larger than the average.

In any case, given that concentricity is often a critical requirement, it will be evident that a sizing operation might be needed to finish the specimens. Presumably, this could be accomplished using one or another of the various sizing methods that are commonly employed by parts makers. However, unlike the typical P/M part, these specimens are near full density and it still remains to be seen how they will react to certain of these methods, particularly, the ones that involve substantial amounts of plastic deformation.

The statistical properties of the four subsets of specimens that are listed relative to this parameter were again consistent with view that the process was stable during the course of the trial. Nevertheless, these data also indicated substantially more variation from subset to subset than was true in the case of either of the earlier discussed properties. Evidently, this property is more sensitive to processing suggesting that it may represent a relatively better means of monitoring the stability of the process than the earlier ones.

The average of the differences between the top and bottom diameters shown in the table is 0.0031 cm (0.0012 ins.). In a great majority of the specimens (e.g. upwards of 90 %), the bottom diameter was the larger of the two. The corresponding standard deviation value was 0.0028 cm (0.0011 ins.). Like the concentricity parameter, it too was almost as large as the average. So, here again, some of the individual values in the distribution were substantially larger than the average value. For example, as shown by the Max value in this case, the largest difference was 3.5 times the average value. In contrast, however, the raw data of the trial also showed that a little over a third of the values were equal to or less than 0.0013 cm (0.0005 ins.). This was not similarly true of the concentricity data.

Experience to date with iron base infiltration has shown that top to bottom differences are generally a consequence of green density variations in the direction of pressing and the frictional forces that exist between the specimens and the sintering trays. Both effect the amount of shrinkage that occurs (in the transverse direction to the direction of pressing) due to sintering in general and particularly, to liquid phase sintering after infiltration. High temperature creep under the influence of gravity is also a possibility but has yet to be unequivocally identified as an actual cause. In any event, in the present case, limited green density distribution studies on randomly selected specimens (i.e. from the 300 that composed the trial series) indicated that while density variations existed, the top to bottom differences were minimal (e.g. less than $\sim 0.03 \text{ g/cm}^3$). Thus, the present differences are thought to be due mainly to the indicated friction effects and liquid phase sintering after infiltration.

Two obvious possibilities exist to eliminate or, at least, minimize these differences. One is to prevent significant liquid phase sintering after infiltration and the other is to reduce the indicated frictional forces. To date, all efforts to prevent significant liquid phase sintering after infiltration in specimens of the present infiltrated composition have failed. However, it is known how to modify the composition to effect this result. In the case of the friction forces, trials have yet to be conducted to determine what the best substrate material is in this regard. However, this is a common problem in liquid phase sintering and it is anticipated that a review of the open literature will either directly provide a solution or indicate the best candidate materials to effect one. Of course, there is also the likelihood that if sizing is required to meet a concentricity requirement in a particular application, it will simultaneously have the effect of eliminating any top to bottom differences that may exist as well.

Contrary to the indications of the other properties in the table, the indications of the four subsets of specimens in this case were generally not consistent with the view that the process was stable during the course of the trial. These values indicated the largest relative variations from subset to subset and more significantly, showed decreasing top to bottom differences during the course of the trial. The apparent

trend in the data suggested the possibility of a declining hot zone temperature during the trial. However, there nothing in the balance of the findings to indicate this and since it was also possible that the trend was simply an artifact of the data, it was eventually concluded that the process had evidently been stable. Nevertheless, the possibility of the indicated temperature variation remained as a reasonable suspicion .

Trial 2 Results

The effects on the same four properties as in the earlier trial of infiltrating the base compacts with a lower infiltrant weight and adding a relatively high temperature liquid phase sintering step are shown below in Table 2. Here again, the specimen number was 48 and precisely the same statistics are listed in the table as were shown in the earlier table.

The average value of the metric density shown in the table is 7.47 g/cm³ (i.e. 99 % of the pore free value). Immersion density determinations on a few of the specimens indicated that the actual average was closer to 7.50 g/cm³. However, based on experience, the expectation was that these process conditions would produce the pore free value, so this result was still lower than expected. Here again, an explanation of the shortfall was provided by the infiltrant residue weights. As previously, the value was nearly an order of magnitude greater than the normally observed value. Thus, there was now virtually no doubt as to the existence of some sort of an infiltrant problem

Table 2 - Results of Infiltrating at 1165 °C (2130 °F) With an Infiltrant Weight of 12.5% of the Base Compact Weight Followed by Liquid Phase Sintering at 1193 °C (2180 °F)

Statistical Property	Metric Density	Diametric Dimensional Change	Absolute Δ Between Parallel & Transverse Diameters		Absolute Δ Between Top & Bottom Diameters	
	g/cm ³	% vs green	cm	(inches)	cm	(inches)
Average	7.47	-0.01	0.0057	(0.0022)	0.0131	(0.0051)
Std. Dev.	0.022	0.060	0.0039	(0.0015)	0.0036	(0.0014)
Max.	7.53	0.10	0.0149	(0.0059)	0.0217	(0.0085)
Min.	7.42	-0.11	0.0003	(0.0001)	0.0062	(0.0024)
Δ Max/Min	0.11	0.22	0.0146	(0.0058)	0.0155	(0.0061)
Δ/s	4.8	3.6	3.8		4.3	
<i>Subset Average and Standard Deviation Values</i>						
Set 1 Nos 1 to 12	7.48 0.020	0.00 0.046	0.0066 0.0082	(0.0026) (0.0032)	0.0144 0.0061	(0.0057) (0.0024)
Set 2 Nos 13 to 24	7.48 0.029	-0.03 0.072	0.0046 0.0069	(0.0018) (0.0027)	0.0127 0.0062	(0.0050) (0.0024)
Set 3 Nos 25 to 36	7.46 0.020	0.02 0.054	0.0052 0.0062	(0.0020) (0.0024)	0.0120 0.0075	(0.0047) (0.0030)
Set 4 Nos 37 to 48	7.46 0.014	-0.03 0.057	0.0062 0.0082	(0.0024) (0.0032)	0.0132 0.0083	(0.0052) (0.0033)

The standard deviation of the infiltrated density in this case was 0.022 g/cm³. This was larger than in the first trial but the process conditions in this trial were consistent with the likelihood of greater variations. For example, if all of the densification in the earlier trial is considered to be due to the infiltrated weight gain, then roughly 10 % of the density increase in this trial (i.e. ~ 0.09 g/cm³) was apparently the result of shrinkage due to sintering. Therefore, the overall densification mechanism in this instance was presumably more complex than earlier and would be expected to produce somewhat more variable

results. In keeping with the larger deviation value, the total range of the variations in the data was likewise larger. Thus, as indicated by the Δ Max / Min value in the table, the range in this case was a little less than 1.5 % of the average. As may be recalled, it was about 1.0 % of the average in the earlier trial.

Comparison of the density statistics of the four subsets of specimens that are listed in the both this and the earlier table will show that the present values were slightly more variable than earlier. In addition, the present data also indicated a slight density decrease in the second half of the trial again raising the possibility of a slightly declining temperature during the course of the process. However, as a quick review of the balance of the subset data in the table will confirm, this indication was not repeated in the case of any of the other properties of the study. So, here again, the most plausible conclusion was that the process was reasonably stable during the course of the trial.

The average diametric dimensional change from green shown in the table is minus 0.01%. When combined with the earlier green expansion value, the indicated average change from the die was about 0.07 %. Compared with the relatively larger dimensional change values of the first trial, the present values confirmed the greater sintering densification that was earlier purported to have occurred in the trial. In addition, by ordinary P/M standards, the present values would normally be preferred to the earlier ones. In part, this preference stems from the possibility to use the compaction die to size the parts should sizing be necessary and in part, from the popular view that a low dimensional change will normally result in low part to part variations. However, in spite of the low values in this case, the present standard deviation value, at 0.060 %, was about 20 % higher than in the first trial. Evidently, as earlier speculated, the greater sintering contribution in this case complicated matters relative to the first trial and led to higher rather than lower part to part variations. Nevertheless, as a practical matter, the present standard deviation value was still reasonably low. More particularly, it indicated specimen to specimen diametric variations which, in absolute terms, were not very much larger than the ones that were observed in the earlier trial. For example, the infiltrated diameter data in this case produced a standard deviation value of 0.0026 cm (0.0010 ins), a Δ Max / Min range value of 0.0096 cm (0.0038 ins) which indicated an average spread about the mean of ± 0.0048 cm (± 0.0019 ins). As may be recalled, the latter values in the earlier case were ± 0.0045 cm (± 0.0018 ins).

The statistical properties of the dimensional change values of the four subsets of specimens in this case showed about the same variation from subset to subset as in the earlier trial. As mentioned, there was no indication of a trend in the data. Thus, as previously, the indication was that the process was stable during the course of the trial.

The average of the differences in the parallel and transverse diameters shown in the table is 0.0057 cm (0.0022 ins.). This result was about 30 % higher than the comparable result of the earlier trial and presumably, reflected the sintering differences in the two trials. However, in comparison with the magnitudes of the changes in the other properties that were apparently attributable to the sintering differences, including especially, the dimensional change and, as will be seen, the top to bottom differences, this particular change was relatively modest. Moreover, a review of the data in the two tables will show that most of the indicated increase occurred in the specimens of the first and fourth subsets. By comparison, this was not similarly true of any of the other properties. The implication was that while sintering was undoubtedly involved in determining the value of this property in both trials, it was not the only factor and, as a general matter, was very possibly not the most important factor.

Accordingly, as shown in an earlier paper, there are several factors other than sintering that affect the final dimensions of a sintered part (10). Briefly, these include the alloy content, the thermal expansion and contraction characteristics, the heating and cooling rates, the temperatures of the alpha to gamma transformation on heating and of the reverse transformation on cooling and (last but not least) the

associated density changes in both cases. Of these, extensive dilatometric experience has generally shown that the triaxial dilations of the density changes that accompany the allotropic transformations in sintering are virtually never isotropic. More significantly, the magnitudes of the dimensional change differences that they produce in the different directions of the specimens are not unlike those that were seen in both of the present trials. Thus, it is strongly suspected that it is these changes that are mainly responsible for the observed differences in the parallel and transverse diameters of the specimens in both trials. As a matter of interest, these effects are known to be sensitive to processing in general and to sintering in particular but the connections, in both cases, are often very subtle.

The average of the differences between the top and bottom diameters shown in the table is 0.0131 cm (0.0052 ins.). This was upwards of 4 times the value that was observed in the first trial. In addition, whereas about 10 % of the top diameters of the specimens in the earlier trial were the larger of the two, in this trial, the bottom diameter was the larger in every case. The corresponding standard deviation value was 0.0036 cm (0.0014 ins.). This was also larger than in the first trial but was nevertheless relatively modest in comparison with the increase in the average value. The respective minimum and maximum values were 0.0062 cm (0.0024 ins.) and 0.0217 cm (0.0085 ins.) yielding a range which was about 4.2 standard deviations in width.

Finally, unlike the indications of this property in the first trial, the values of the four subsets of specimens in this trial were consistent with the view that the process had been stable during the course of the trial. The differences in the values from subset to subset were relatively modest and there was no indication of a trend in the data.

Surface Appearance

The infiltrated surface appearances of the specimens of both trials were problematic. As expected, the infiltrant slugs had left thin, centrally located residues which were roughly circular in shape and ~ 2 cm (0.75 ins) in diameter. Unexpectedly, however, the residues were somewhat adherent to the underlying surfaces and had to be mechanically removed. This was done manually using a knife edge as a pry. In the case of the specimens of the first trial, the residues normally came off the surfaces intact and without too much difficulty. In the case of the specimens of the second trial, there was a greater tendency for the residues to breakup during the process and they were generally more difficult to remove. These changes evidently reflected the effects of the generally higher process temperature in the second trial.

The general condition of the un-infiltrated surfaces of the specimens as well as of the infiltrated surfaces to within a few millimeters of the residues was excellent. In comparison, the condition of the surfaces under and just adjacent to the residues was poor. In the general case, these areas were either flawed with erosion pits or covered with residual particles including partially melted and/or unmelted iron and residual oxides, sulfides and possibly, un-reacted graphite. In some places, the pitting and particles occurred together. In the case of the specimens of the first trial, low power microscopic examinations suggested that both defects were more cosmetic than structural. However, this was not true of the specimens of the second trial. The erosion pits in particular, in this case, appeared to be deep enough in many places to threaten the structural integrity of the surfaces.

Neither these findings nor the aforementioned high residue weights were expected. Earlier, albeit more limited, studies had generally produced very different results. The observed infiltrant residue weights (as a percentage of the starting weights) normally varied somewhat but were never seen to be as high as the present ones. Similarly, although the resultant infiltrated surfaces were seldom flawless, they were generally better than the present ones.

The simplest explanation of these findings was that they were a result of the slight changes that were made to the infiltrant composition. As will be recalled, these included the elimination of an heretofore standard zinc stearate addition and a minor increase in the refractory oxide content of from 0.035 % to 0.050 %. As between the two, the removal of the stearate was regarded as the potentially more relevant one in terms of explaining the findings. For example, earlier work had shown that both high residue weights and poor surfaces could be the result of graphite inhomogeneities in the infiltrant which pointed directly to the stearate. As mentioned, it had originally been included in the composition specifically because of its beneficial effects in helping to homogenize the graphite.

Trial 3 Results

The trial was designed primarily to examine the effects on the residue weights and surface appearances of three modifications of the base infiltrant of the study (i.e. the infiltrant of the first two trials); the standard of comparison being the base infiltrant itself. The modifications included re-instating the zinc stearate into the composition plus two consecutive increases in its refractory oxide content of 0.025 % each. Thus, the trial essentially involved comparing the behaviors of four different infiltrants. The specific compositions in each case and the names used to identify them in discussing the results are shown below in Table 3. As may be recalled, the processing that was used in this trial was similar to but not precisely the same as that of the first trial.

Table 3 - Trial 3 Infiltrant Names And Compositions*

Infiltrant ID	Graphite	Silicon	Refractory Oxide	Zinc Stearate
	%	%	%	%
Base	4.4	0.2	0.05	-
Base + ZnSt	4.4	0.2	0.05	0.1
Base + ZnSt + 0.025 Oxide	4.4	0.2	0.075	0.1
Base + ZnSt + 0.050 Oxide	4.4	0.2	0.100	0.1

* Iron and impurities composed the balance of the compositions in each case .

The infiltrated surfaces of one each of the two specimens that were infiltrated with the four compositions are presented in Figure 1 overleaf. The resulting infiltrant residues in each case are also shown in the figure.

Superficially, the surfaces of the specimens in the figure were typical of the surfaces of the majority of specimens of the first two trials. Upon closer inspection, the surface of the specimens infiltrated with the Base infiltrant were about the same as those of the first trial and, as mentioned, appreciably better than those of the second trial. In comparison, the surfaces of the specimens infiltrated with the three modified infiltrants were marginally better than those of the Base infiltrant but still not as good as seen in earlier trials and therefore, not entirely representative of what the technology has to offer in this respect.

As previously, low power microscopic inspections of the surfaces generally showed that the residue affected areas were largely, if not completely, covered with residual particles and otherwise sporadically flawed with erosion pits. At a magnification of 10 x, it was evident that the residual particles were small agglomerates of varying size. They appeared to be largely composed of unmelted iron and admixed oxides but must have also contained the un-melted impurities of the iron that were deposited by the passage of the liquid phase of the infiltrant during the infiltration process. Based largely on their dark color, the particles may have also contained unreacted graphite. However, this was uncertain since both iron and its impurities are also typically dark in color. As indicated by the odor that came off the surfaces, sulfides were present as well. As a general matter, the particles per se did not appear to represent more than a cosmetic nuisance

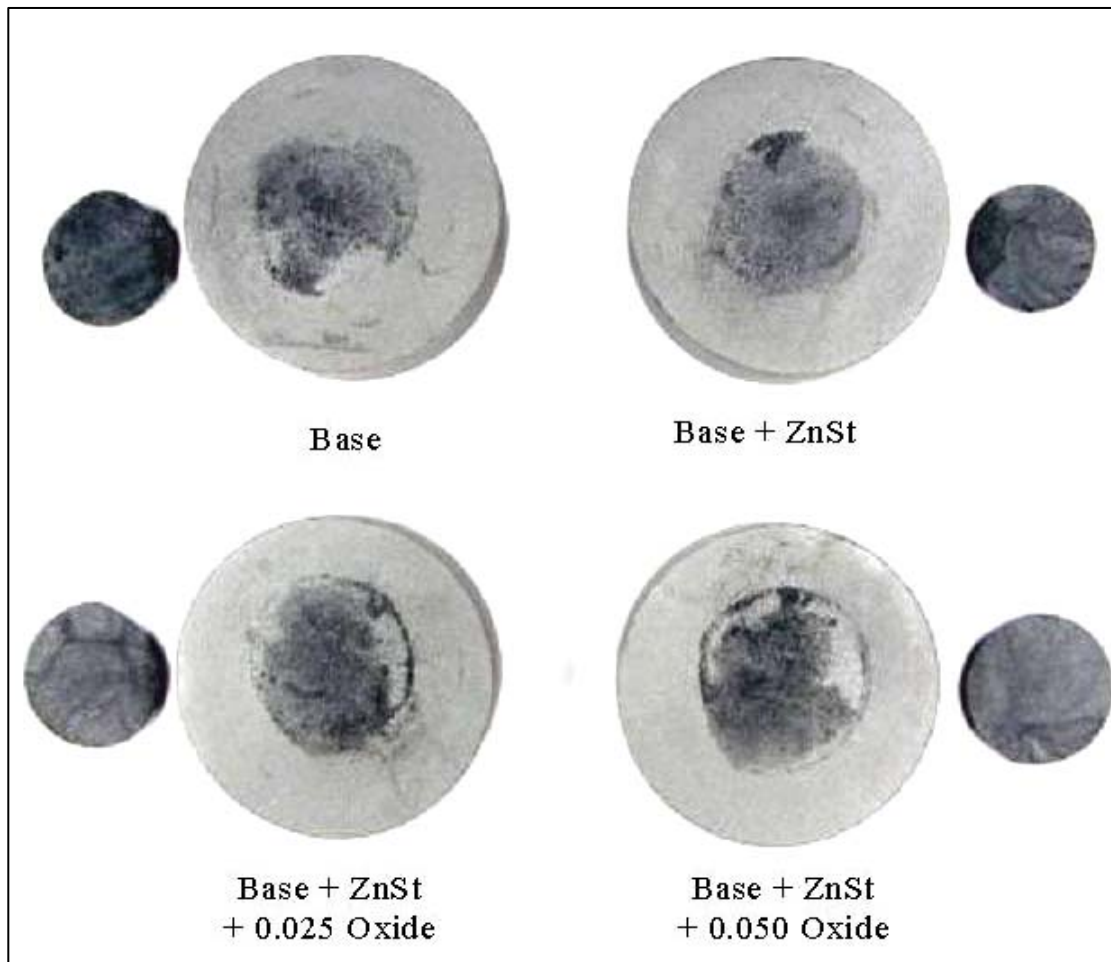


Figure 1 - Infiltrated surfaces and residues of Trial 3 specimens.

The low power microscopic inspection also revealed more about the erosion pitting of the surfaces. The pits in the surfaces of the specimens that were infiltrated with the Base composition were about like those of the specimens of the first trial. In contrast, the pitting of the surfaces of the specimens infiltrated with all three of the modified compositions was noticeably lighter both in terms of depth and frequency of occurrence. However, there was not much difference in this respect between the three. The implication was that the zinc stearate had been effective to improve the graphite uniformity of the mixes and in fact, was much better in this respect than the metallic soap of the oxide. Here again, the pitting in all four cases appeared to represent little more than a cosmetic nuisance. Nevertheless, its not inconceivable that they could pose a problem for highly critical applications.

The infiltrant residues that are shown in the figure were also examined both visually and microscopically. As earlier, the residues in all four cases were again somewhat adherent to the underlying surfaces and had to be removed with the help of a knife edge. However, they were otherwise easy to remove and as is clear from the figure came off the surfaces intact. Notice that the residues in the top of the figure (i.e. corresponding to the Base and the Base + ZnSt infiltrants) are slightly smaller in diameter than those in the bottom of the figure (corresponding to the infiltrants that contained the extra oxide units). As will be shown later, this was because the residue weights were correlated with the oxide content of the infiltrant composition and generally increased with increase in its content.

All four of the residues contained remnants of the liquid phase. However, whereas this was very apparent in the case of the larger diameter residues, it was barely discernible in the case of the smaller ones. Evidently, the capillary forces of the base compact, which are generally known to be responsible for drawing the liquid phase of the infiltrant into the compact, were counter balanced by forces of a similar origin that developed in the oxide matte as it formed in the wake of the infiltrating liquid. Apparently, as the oxide content of the infiltrant increased, the size of the resultant matte increased as well with the result that it retained more of the liquid phase of the infiltrant. Since there was little, if any, attendant improvement in the associated surfaces, it was evident that oxide contents in excess of the present 0.05 % and very possibly, the earlier 0.035% as well, were counter indicated.

The effects which this behavior had on the density and diametric dimensional change properties of the infiltrated specimens are shown below in Table 4. In addition to the density and dimensional change, the table also lists the residue weight, the residue weight as a percentage of the base compact weight and the infiltrated weight (i.e. the weight of the infiltrant that evidently infiltrated the compact) as a percentage of the base compact weight. The values in the table represent the averages of the results of the two specimens that were processed in each case.

Table 4 - Density and Diametric Dimensional Change Effects of the Trial 3 Infiltrants

Infiltrant	Infiltrated Density	Dimensional Change vs Green	Residue Wgt	Residue Wgt vs Base Cp Wgt	Infiltrated Wgt vs Base Cp Wgt
	g/cm ³	%	grms	%	%
Base	7.40	1.08	1.2	0.8	14.9
Base + ZnSt	7.40	1.08	1.3	0.8	14.9
Base + ZnSt + 0.025 Oxide	7.40	0.94	1.9	1.2	14.5
Base + ZnSt + 0.050 Oxide	7.39	0.76	2.6	1.7	14.0

As indicated above, these data show that the residue weight increased with increase in the oxide content of the infiltrant. Interestingly, however, the data also showed that the final densities of the specimens were essentially all the same in spite of the indicated liquid losses to the residues. Apparently, liquid phase sintering after infiltration as manifest in decreased dimensional change values compensated for the losses. In fact, according to the data, the dimensional change is linearly correlated with the residue weight as well as with each of the two other parameters in the table that reflect it. As may be easily verified, the relationship is virtually a perfectly straight line in each case.

The evident implication of these findings is that the dimensional change can be manipulated over a fairly wide range of values without adverse affect to the infiltrated density simply by changing the infiltrant weight. Thus, a potential advantage of the technology is the possibility to fine tune the parts making process by using the infiltrant weight to counteract the natural variations that occur from time to time in the less controllable parameters that are involved.

Finally, the obvious general indication of both these findings and the surface appearance results is the need of additional development work to improve the performance of the infiltrant and especially, that of the oxide addition with regard to how it affects both the nature of the residue and the integrity and appearance of the resulting surface.

SUMMARY AND CONCLUSIONS

The present study examined the factors effecting the dimensional change characteristics and surface quality of iron base infiltrated parts. The objective was essentially to define the conditions needed to implement the process as a practical matter.

It was explained at the outset that the infiltrated compositions according to the present technology offered a wide choice of dimensional change possibilities ranging from a high of upwards of 1 % to a low of 0 % or less. However, it was uncertain what effects the processing required to produce a particular value in this range would have on the dimensional consistency of the resulting parts. Thus, the primary objective of the study was to determine which of the extremes of the indicated range was associated with the best overall dimensional control in terms of part to part variability and within part uniformity.

It was also explained at the outset that while the infiltrated surface appearance of parts according to the technology is dependent on processing, it is largely determined by the infiltrant composition. Earlier work had shown that purposeful additions of a refractory oxide to the infiltrant were especially effective to improve the surface but it still remained to optimize the composition with respect to the oxide. As a consequence, the infiltrant composition that was used in the dimensional change determinations was a minor modification of an earlier existing one. The modification included the removal of a small zinc stearate addition and a slight increase in the oxide content. As it turned out, this composition did not perform as well as expected. The surface quality of the resulting parts was not as good as earlier and the infiltrated densities were relatively lower as well. Thus, a very limited trial was included in the study to determine the effects of re-instating the stearate addition and of further increases in the oxide content of the infiltrant.

Two trials were conducted to evaluate the indicated dimensional change effects. The base compacts in both cases were cylindrical discs measuring about 4.5 cm (1.75 ins.) in diameter and 1.4 cm (0.55 ins.) in height. In addition to the infiltrated density and dimensional change properties, diametric parameters that indicated the concentricity and the top to bottom uniformity of the specimens were also evaluated. The values of selected statistics of four subsets of the specimens that reflected the order of processing in the trial were compared to assess the operational stability of the processing.

In Trial 1 of the study, the specimens were processed at a low temperature ($1165\text{ }^{\circ}\text{C} \approx 2125\text{ }^{\circ}\text{F}$) and using a high infiltrant weight (i.e. 16.5% of the base compact weight). The resulting infiltrated density averaged 7.40 g/cm^3 versus an expected value of $\sim 7.47\text{ g/cm}^3$ and a pore free value of 7.54 g/cm^3 . The shortfall in density was traced to relatively high infiltrant residue weights. The standard deviation was 0.018 g/cm^3 and the corresponding range of variation in the data was about 1% of the average. The average diametric dimensional change was 1.29 % from green and 1.37 % from die. The standard deviation in this case was 0.051 % and the corresponding 4σ value of 0.204% indicated a maximum diametric variation about the average of $\pm 0.0046\text{ cm}$ (0.0018 ins.) which the diameter data of the trial confirmed. The concentricity parameter indicated that the specimens were out of round by an average of 0.0043 cm (0.0017 ins.). The top to bottom uniformity parameter indicated that the bottom diameters were larger than the top ones in 90% of the cases. The average difference was 0.0031 cm (0.0012 ins). The general indications of the data of the four subsets of the specimens was that the process was stable during the course of the trial.

In Trial 2, the specimens were initially processed at the same low temperature as in Trial 1 but using a relatively lower infiltrant weight (i.e. 12.5 % of the base compact weight) and then submitted to a high temperature liquid phase sintering step at $1190\text{ }^{\circ}\text{C}$ ($2180\text{ }^{\circ}\text{F}$). The infiltrated density averaged 7.47 g/cm^3 versus an expected value of 7.54 g/cm^3 (i.e. the pore free value). Here again, the shortfall was traced to relatively high infiltrant residue weights. The standard deviation was 0.022 g/cm^3 and the corresponding

range of variation in the data was about 1.5 % of the average, 50% greater than in Trial 1. The average diametric dimensional change in this case was -0.01 % from green and 0.07 % from die. The standard deviation was 0.060 %, about 20 % greater than in the first trial. The corresponding 4σ value of 0.24% indicated a maximum diametric variation about the average of $\pm 0.0053\text{cm}$ (0.0021 ins.). The actual diameter data of the trial indicated a slightly lower variation of $\pm 0.0048\text{cm}$ (0.0019 ins.). Here again the concentricity parameter indicated that the specimens were out of round. The average was 0.0057 cm (0.0022 ins.), about 30 % greater than in the first trial. The top to bottom uniformity parameter indicated an average difference of 0.0131 cm (0.0051 ins.), a little over 4 times the value of the first trial. In this case, the bottom diameters of the specimens were uniformly larger than the top ones. The largest difference in the data was 0.0217 cm (0.0085 ins.). As earlier, the general indications of the data of the four subsets of the specimens was that the process was stable during the course of the trial.

In view of these findings, it was concluded that at least in the near term, there is essentially nothing to be gained in terms of dimensional consistency from using the sinterability inherent in the compositions of the technology to produce a low average dimensional change value. The potential advantage that a near zero dimensional change offers to use the compaction die to size the parts would almost certainly be negated in this case by the very large top to bottom differences that were observed. In addition, the results of earlier studies suggested that the slightly higher infiltrated densities that the associated processing is likely to effect will generally not lead to significantly higher tensile properties. In part, this is because the pores that remain after infiltration are both closed and generally well rounded and in part, because at contents of 1 or 2 %, they are not the dominant stress raising feature of the microstructure. Accordingly, the so-called 'compacted' graphite precipitates that normally occur in the microstructures of the subject compositions are typically more angular and present in much higher concentrations (e.g. ~5 % or more).

The condition of the infiltrated surfaces of the specimens of both trials was not as good as expected. The infiltrant residues were somewhat adherent to the surfaces and had to be mechanically removed. The residue affected areas were generally covered with residual particles and otherwise randomly flawed with erosion pits. Both defects appeared to be reasonably innocuous in the case of the specimens of the first trial but were potentially problematic in the case of the specimens of the second one.

These findings combined with the high infiltrant residue weights that were also observed in both trials indicated an infiltrant problem and led to the third trial of the study. The results of this trial indicated that restoring the small zinc stearate addition to the infiltrant decreased both the incidence and average depth of the erosion pits but had little to no effect in decreasing the residual particle coverage or the residue weight. In comparison, the concomitant increases in the oxide content of the infiltrant apparently had no effect on either the pitting or the residual particle coverage but did result in further increases in the residue weight. Interestingly, the potential density decreases attending the liquid losses to the residues were just compensated in each case by liquid phase sintering after infiltration. Thus, the final infiltrated densities were unaffected but the average dimensional change of the specimens decreased accordingly. The implication was that the infiltrant weight could be used as a practical matter in parts making as a simple expedient to control the average dimensional change value.

Otherwise, it was concluded that the zinc stearate should be incorporated as a permanent (standard) component of the infiltrant and that more research is needed to determine both the best oxide and oxide content to be used in the composition.

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