PROPERTIES OF PARTS MADE FROM A BINDER TREATED 0.45% PHOSPHORUS CONTAINING IRON POWDER BLEND

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ABSTRACT

Studies were conducted to determine the effects on property variability of parts made from a binder treated blend. The blend was a lubricated admixture of Fe_3P and Ancorsteel 1000B iron powders. The parts were cylindrical bushings having a nominal wall thickness of 0.25 inches and otherwise measuring 1.5 inches in outside diameter and 2.0 inches in height. In conducting the study, an analysis of variance design was employed to enable assessment of the relative contributions of six variance sources as follows: 1) testing; 2) microsegretation; 3) part to part for pairs pressed back to back and sintered side by side; 4) sintering within trays; 5) sintering tray to tray, and 6) macrosegregation.

Relative to parts made from a companion control blend, the results of the study showed that the binder treatment was effective in reducing variability in each of the following properties: dimensional change, crush strength, hardness and average phosphorus content. The Analysis of Variance results suggested that the observed reductions were due primarily to reductions in sintering was also indicated to be a significant variance source, although in this case, it also appeared to be affected by the presence of microsegregatlon.

The findings are assessed and discussed both in traditional statistical terms and in terms of Statistical Process Control. The latter terms are employed to show how the variability reductions may be translated into quality and/or economic benefits in actual parts making situations.

INTRODUCTION

Compositional variations in premixes by virtue of particle segregation and dusting phenomena are fairly well known in the

P/M Industry. Such effects are especially apparent in high alloy compositions but are present to some extent in all admixtures regardless of alloy content. In the case of dusting, visual indications are a common daily occurrence. In the case of segregation by particle migration, visual indications are less frequent but no less well known. Almost anyone reasonably experienced in the art can cite at least one instance of such phenomena.

Compositional variations associated with segregation and dusting are also manifest in variations in the physical and mechanical properties of both the blends and the parts which are made from them. However, in as much as segregation and dusting are difficult to quantify and reproduce, the relation of cause and effect in these regards is known at best only qualitatively and in many instances is more a matter of speculation than actual fact. Nevertheless, an imposing, albeit largely empirical body of evidence exists to suggest that segregation and dusting exact both technical and economic penalties of the industry. Technically, for example, these phenomena limit the application of the method of preparing alloys by simple admixing both as to alloy content and alloy type.(1,2,3) In regard to economics, segregation and dusting increase costs in a variety of ways. The most well known of these include part rejections due to unacceptable dimensional variations and the attendant productivity losses associated with efforts to reduce or prevent such rejections. (4,5,6)

Various powder manufacturing techniques are available to the industry which either completely avoid or greatly diminish the adverse effects of segregation and dusting. The three most noteworthy are: Preparation of prealloys by atomization (7); Diffusion bonding by annealing (8,9) and, Binder treatment of premixes.(1O,ll) Of the three, the binder treatment method is the least well developed, especially as regards the basic ferrous industry.

Research in Hoeganaes Riverton Laboratories in the last several years has been directed to altering this particular situation. As of the present time, first stage development of a practical binder treatment process is all but complete and initial scale up to production size is in progress. In addition, a number of parts manufacturing studies aimed at quantifying the effects of the treatment are either in progress or have been completed. Some of these studies are laboratory size in scale and some, generally the more recent ones are production size. Various premix compositions as are typically specified in the industry and containing one or more of the common admix ingredients including graphite, copper, nickel and ferrophosphorous were included in the studies. The purpose of the present paper is to report the findings of one such study in which the principal alloy admix ingredient was ferrophosphorous.

EXPERIMENTAL PROCEDURE

The study was laboratory size in scale. Its basic objective was to assess the potential of the binder treatment method. The general scheme employed was to process a binder treated mix and a regular mix of the same composition under nominally the same conditions and compare the properties of the resultant parts as to statistical variability. The part geometry studied was a cylindrical bushing and the effects on six properties were determined: sintered density, the O.D.. and I.D. dimensional changes, hardness, crush strength, and phosphorus content. Since property variability may arise in several ways, the Analysis of Variance method was employed to enable an assessment of the relative contributions to the findings of testing, processing, and segregation. The particulars of this method as well as of those of the other procedures employed are detailed below.

PREMIX PREPARATION

The nominal premix composition was 0.45% phosphorus, 0.75% Acrawax C, 0.25% zinc stearate, and balance iron. The phosphorus was added as Hoeganaes AB Fe_3P and the iron as Hoeganaes Ancorsteel 1000B.

The regular mix was made by blending the ingredients in a double cone blender for 1/2 hour. The binder treated mix was made by a proprietary process. The binder, also proprietary, was a solid organic material and was added in the amount of 0.125% by weight. Both mixes were made to a weight of 500 lbs. and both were packed out in 100 lb. increments.

PRELIMINARY TESTING

The two mixes were tested in advance of any actual parts making as a quality control measure. Both green and sintered properties were determined. The determinations were generally conducted in accordance with P/M Industry standards. Sintering was at 2050°F for 30 minutes at temperature in dissociated ammonia. The green density in advance of sintering was 6.8 g/cm^3.

DUSTING RESISTANCE TESTING

In addition to the commonly measured properties, the preliminary tests also included determinations of the phosphorus dusting resistances of the two mixes.

In general, the so-called dusting resistance property has value both to assess the effectiveness of the binder treatment processing and to indicate the relative segregation and dusting tendencies of any admix ingredient of interest. The particulars of the method and the apparatus used to conduct the test were briefly as follows.

The test consists of elutriating a sample of the subject mix with nitrogen under controlled conditions of flow rate and time. For the studies reported here, the sample size was typically 20 to 25 grams and the flow rate and time were consistently 2 liters per minute for 15 minutes. The test apparatus consists simply of a cylindrical glass column vertically mounted on an Erlenmeyer flask. The glass column 5 equipped with a 400 mesh screen plate to support the sample and the flask has a side inlet to emit the nitrogen. The dimensions of the glass column are 7 inches in length and 1 inch in inside diameter. The screen plate is positioned 1 inch above the mouth of the Erlenmeyer flask. The flask is of 2000 ml capacity;

The parameter which is of interest to assess the dusting tendency of a particular ingredient of a mixture is the ratio as determined by chemical analyses of the ingredients content after the test to that before. This value is typically expressed in percent and is referred to as the ingredients dusting resistance.

As a matter of interest, the test has been in existence since early 1983 and studies have shown that when properly conducted it yields reproducible results for all of the common alloy ingredients typically used in iron powder mixtures.

PART GEOMETRY AND COMPACTION PROCEDURE

The part was a cylindrical bushing nominally measuring 1.5 inches in outside diameter, 1.0 inch in inside diameter, and 2.0 inches in height. Compaction was to a density of 6.8 g/cm^3 which yielded an average part weight of about 0.48 lbs.

The compaction was done on a Dorst TPA 50 at A.C. Compacting Presses, Inc. located in North Brunswick, New Jersey. To make the indicated part the press was set up as follows.

Press Position:	2.5″
Fill Position:	4.125″
Prepress:	0.250″ to 0.312″

In setting up the press, density checks were made which suggested that the resulting parts would be reasonably symmetrical end to end. The pressing rate employed with both the regular and binder treated mixes was 10 parts/minute.

Owing to the facilities available in connection with the press, the press hopper had to be charged manually. The hopper was about 16 inches in depth and had a capacity of about 175 lbs. These facts combined with the necessity to charge manually led to the speculation that unless special procedures were employed, it would be impossible to reproduce the charging conditions from mix to mix or for that matter, even from charge to charge within a mix. In particular, there was a concern that unless precautions were taken, significant extraneous variations might be introduced which could easily lead to spurious results and very possibly the wrong conclusions. Consequently, prior to pressing the subject mixes, a study was conducted in an effort to develop a reasonably reproducible charging practice.

The resultant practice was as follows. A special powder charging funnel was constructed to fit over the mouth of the press hopper. The particular advantage of the funnel was that its design allowed the operator to minimize the free fall distance from the packing container to the funnel and thus enabled him to control the discharge of the powder to the funnel. Once in the funnel, of course, the powder discharged to the press hopper under the influence of gravity and in accordance with its own flow characteristics. Further advantages of the funnel were that it centered the powder stream and covered the mouth of the hopper so as to minimize dusting losses.

In addition to the use of the funnel, it was also found necessary in order to obtain reasonably reproducible discharge of the powder from the hopper to the press to charge the powder in measured increments and to suspend the pressing operation during charging until the powder was completely transferred from the funnel to the hopper. Based on the hopper capacity and the mix weight, the charge increment selected to meet this requirement was 100 lbs.

When actually pressing the subject mixes, the first 100 lbs. of mix was used to allow the tools to approach thermal equilibrium and to zero in on the aim density, and height, i.e. 6.8 g/cm^3 and 2.000 inches. Most of the resulting parts were discarded although a few were held for potential later use in preliminary trial work. Subsequently, in making the parts for the primary studies, the pressing operations were controlled by measuring the weight and dimensions of every 25th part and by making pressure and/or fill adjustments as needed to maintain the density to within ± 0.02 g/cm^3 and the height to within ± 0.003 inches of the aim values. A log was kept of all such measurements and adjustments as well as associated comments so that a complete record of the pressing operations would be available for later referral.

In order to satisfy certain procedural requirements as described below, it was necessary to retain knowledge of the parts pressing sequence. This was accomplished by packing the parts out as they came off the press to containers having numbered cubicles. In addition, it was also considered necessary to retain knowledge of the orientation of the parts during pressing. This was accomplished by designing the top punch of the tools used for the study with a small indentation and subsequently by recording the position of the indentation once the tools were in the press.

Only 300 of the original 500 lbs. of mix in each case were actually pressed to parts. This included the 100 lbs. used in initiating the operations. Thus, with an average part weight of 0.48 lbs. the yield in terms of parts available for further processing was about 400 per mix. Based on a decision made during the course of the work, the remaining 200 lbs. in each case were set aside for a separate purpose.

As will be seen, the number of parts which were actually used in the balance of the study was considerably less than the numbers available from the pressing operations. This was due in part, of course, to the practical necessity to limit the size of the trial to what could reasonably be done in terms of testing. However, independently of any concern for test load, it was thought prudent to hold a substantial number of parts in reserve as a store against potential future needs For example, there was the possibility of the need or the desire to look at green properties at some point as well as the likelihood of future interest in sintering conditions other than the ones presently to be described.

SINTERING EQUIPMENT AND GENERAL PROCESS DETAILS

The sintering step was carried out in a Drever furnace at Drever Company headquarters located in Huntingdon Valley, PA. The furnace was a 10 inch belt P/M type furnace which was reportedly only used for R&D purposes. Its general plan is shown in Figure 1.

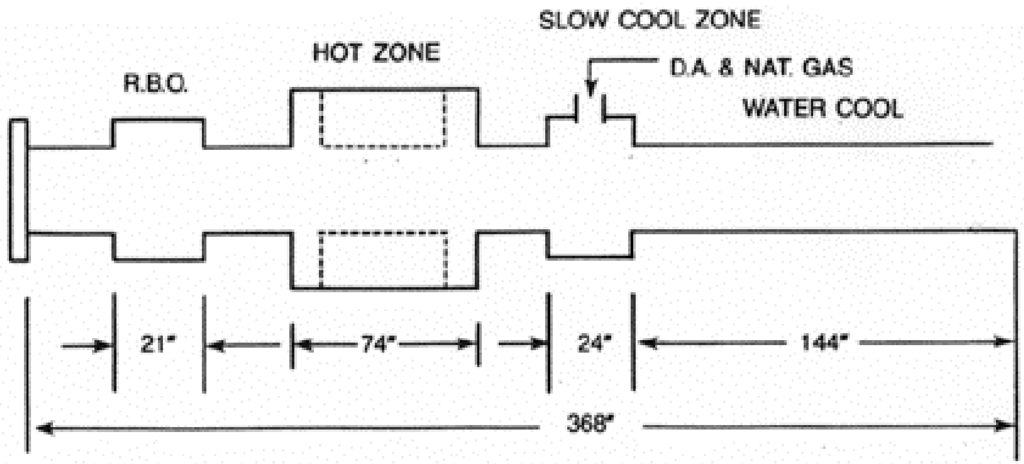

Figure 1 - Plan View of Sintering Furnace

Apart from the features shown in the figure, the furnace was operable either continuously or in the batch mode and was otherwise equipped with sintering trays. The plan was to sinter continuously and as will be seen to employ special parts arrangements. Consequently, it was decided to use the sintering trays since under the indicated circumstances, they were especially convenient to load and unload the belt.

The aim sintering conditions were 2050°F for 30 minutes at temperature in dissociated ammonia. A series of preliminary studies were conducted to determine the furnace settings, atmosphere flow rate and tray spacing needed to achieve these conditions for the furnace loading and belt speed of interest. The results of these studies are indicated in Figure 2. The temperature profile typical of the furnace settings which were eventually used in the study is shown in the figure and the values employed in connection with the other process parameters involved are also listed.

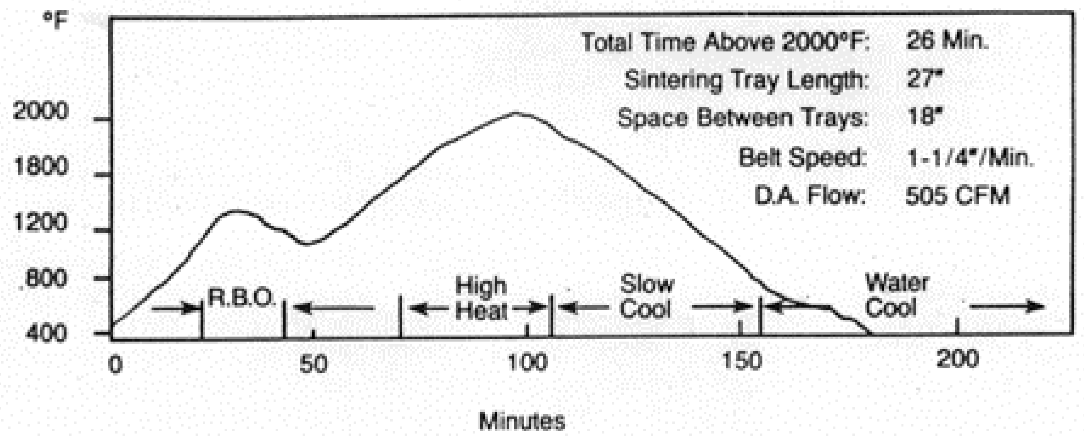

Figure 2 - Furnace Temperature Profile and Sintering Details

EXPERIMENTAL ASPECTS OF THE SINTERING PROCEDURE

The experimental aspects of the sintering procedure were regarded as including:

1) The number of parts to be sintered.
2) The selection of the parts from the total number of parts available; and,
3) The arrangement of the parts during sintering.

The decisions taken in regard to these aspects and the reasons underlying the decisions are outlined below.

Number For Sintering

The actual number of parts eventually submitted to sintering was less than 15% of the total number available. This included those needed for various preliminary studies as well as for the primary study. The precise number used in the primary study was 48 per mix. This number was first suggested by the size of the sintering trays. However, the actual decision to use it was made mainly on the basis of statistical considerations. Without going into details, these considerations led to the conclusion that a sample size of 48 would be adequate to estimate variability with the precision needed to indicate small but physically significant variability differences.

Selection For Sintering

The selection of the particular 48 parts from the 400 or so available with each mix was perhaps the single most important aspect of the entire procedure. However, since as explained below it was necessary to treat the selection as an experiment in itself, it was also perhaps the weakest aspect. The objectives of the selection, of course, were both to represent the two mixes fairly and to show the effects of the differences in the two. The difficulty was that because of the relative novelty of the study, there was no way of knowing in advance how best to achieve these objectives or, in fact, whether they were even mutually compatible. For example, it was reasonable to expect that there should be effects from differences in segregation tendency but it was not known whether these effects would be manifest in pressing or sintering or both or where in the parts pressing sequence they would be most likely to be observed or finally whether they would necessarily be found at the same points in the two mixes. In addition, it was not known whether segregation effects would be the only effects. In particular, as will be seen later, the binder treatment method has pronounced effects on powder flow behavior as well as on segregation tendency.

Consequently, in view of the unknowns, it was necessary to be somewhat arbitrary and after due consideration it was decided to select the parts in accordance with the following guidelines:

- To base the selection on the parts pressing sequence and to select the same parts in terms of sequence for each mix.

- To minimize the likelihood of introducing extraneous effects due to pressing by limiting the selection to the product of a single pressing run of 100 lbs. of mix and further to the particular run in each case which exhibited the least need of density and/or height adjustment.

- To devise a selection which offered the possibility of later separating the effects of pressing, sintering, and segregation by application of the Analysis of Variance method.

Implementation of these guidelines led to the selection shown in the matrix in Figure 3. The matrix indicates the parts in accordance with their numerical sequence in pressing. Review of the numbers in the matrix will show that the selection was composed of six groups of eight parts each. The groups are separated end to end by an interval of twenty-five. The eight parts composing a group are in sequence and as shown in the matrix are arranged in pairs.

Group	Pair			
	1	2	3	4
1	17,18	19,20	21,22	23,24

2	42,43	44,45	46,47	48,49
3	67,68	69,70	71,72	73,74
4	92,93	94,95	96,97	98,99
5	117,118	119,120	121,122	123,124
6	142,143	144,145	146,147	148,149

Figure 3 - Parts Selected for Sintering

It will be convenient to what follows to identify a particular part pair using a three digit code which indicates the mix type and the position of the pair within the matrix as follows:

A: Mix Type: R for regular; and B for binder treated
B: Group Number, e.g. 1 to 6
C: Pair Number, e.g. 1 to 4

Thus, for example, R42 refers to parts 94 and 95 of the regular mix and B54 to parts 123 and 124 of the binder treated mix.

Arrangement For Sintering

The parts were arranged for sintering in a highly specialized manner. The objective was both to ensure similarity of treatment as regards the two mixes as well as to obtain specific information on the effects of sintering.

The arrangement was developed around part pairs rather than individual parts and is indicated in Figure 4 in terms of the three-digit codes as outlined above. Review of the figure will show that the arrangement was over two trays and was symmetrical with respect to the two mixes. Each tray contained twelve pairs of each mix. The positions assigned to the pairs within the trays were determined by a random selection scheme. Closer inspection of the figure will further show that the six groups indicated in the parts selection matrix were evenly distributed between the two trays. Each group was represented by four parts or two pairs in sequence in each tray.

R32 G32	G62 R62
R31 G31	G61 R61
R41 G41	G22 R22
R42 G42	G21 R21
R11 G11	G12 R12
R52 G52	G51 R51

R23 G23	G13 R13
R64 G64	G44 R44
R33 G33	G54 R54
R34 G34	G24 R24
R53 G53	G14 R14
R63 G63	G43 R43

Tray 1 Tray 2

Figure 4 - Parts Arrangement for Sintering

In sintering the parts, an effort was made to prevent end effects due to imbalances in furnace load by bracketing the trial trays by fully loaded trays of parts saved of the discard from the pressing operations. As a minor point of interest, these same parts were also used in setting up the furnace as described earlier.

ANALYSIS OF VARIANCE DESIGN

The general experimental scheme of comparing parts of the two mixes as made under similar conditions was basically sufficient to accomplish the objectives of the study, i.e. to estimate the potential merits of the binder treatment method. However, a difficulty with the general scheme was that outside of the differences in how the mixes were made there was little if anything in it to indicate the underlying causes of the resultant findings. The danger was that the natural inclination to speculate would lead to misunderstandings as to the real meaning of the findings. With proven technologies, such misunderstandings are often problematical but seldom disastrous. However, the same cannot be said of new technologies. Consequently, it was considered essential to supplement the procedure in some way in order to get a better understanding of the findings. Since variability was the major property of interest, an analysis of variance or so-called ANOVA study was a natural choice in this respect.

Much of the experimental procedure as so far outlined was designed to accommodate the needs of this study. The objective of the present section is to explain how this was accomplished.

The particular ANOVA type selected for the study was what is known in statistical parlance as a nested or hierarchical type.(12) As a general matter, application of the ANOVA method requires a knowledge of the variance sources which contribute to the variability result under analysis and a test program which admits of the possibility of separating the sources either as pure values or less desirably, as composite values of as few components as possible. The difficulty with composite values is that their interpretation is frequently less than straightforward.

In the present case, four basic variance sources were considered to be involved. These were testing, pressing, sintering and segregation. As will be recalled, the plan was to compare the mixes on the basis of the variabilities of six different part properties. From a consideration of the sources and the properties, it was clear that while some of the resulting variance components would be separable as pure components, most would not. In general, although the testing and segregation sources contributed to this result, it was due primarily to the fact that the study was based on sintered properties which, of course, necessarily included the effects of pressing as well. Thus, it was evident that the planned analysis would have to cope with the problem of dealing with a number of composite variance values.

A useful strategy in cases where composite values cannot be avoided is to try to simplify their interpretation by devising an experimental procedure which suppresses variance sources which are not of interest in relation to those which are. The need of this strategy in the present case was recognized from the outset and as explained below, it was extensively employed in developing the procedure as so far indicated.

Of the four variance sources, the testing source presented the least difficulty. It was essentially separable as a pure value for five of the six properties of interest. The required procedure was to obtain at least one replicate set of test measurements in each case. The exceptional property was crush strength which, of course, could not be measured more than once because the test is destructive.

Of the remaining variance sources, there was considerably more interest in sintering and segregation than there was in pressing. Consequently, it was decided to try to suppress pressing variations as much as possible and as will be evident in general but especially from the pressing procedure itself, a very substantial effort was made to implement this decision both in developing and in carrying out the procedure.

As between sintering and segregation, segregation as indicated by alloy content was separable as a pure component without too much difficulty. In the actual ANOVA study which was done in this connection, there were six variance components including testing, microsegregation and four different types of macrosegregation. A description of the latter is best left to later. However, it will be helpful for the present to indicate that microsegregation was regarded as within part variations in composition and macrosegregation as all other compositional variations including those from part to part or more generally, as within mix variations for units of mix of part size or larger.

In addition to segregation per se, there was also interest in its effects. Designing the procedure to achieve a reasonable separation in these regards was perhaps the most complex aspect of the entire study. The particular design features which address this aspect were incorporated in the procedures used both to select the parts and to arrange them for sintering.

The parts selection procedure was developed to a significant extent around two assumptions as follows. One was that the properties of a small number of parts made in sequence should be relatively free of differences due to segregation. Therefore, sintering could be studied reasonably independently of segregation by basing the studies on the properties of such parts. As will be explained in more detail below, the associated variance was divided into three different components. The second assumption was that is should be possible to impose reasonably the same sintering conditions on several groups of such parts by randomizing the parts with respect to the tray positions available in sintering. Therefore, if segregation differences existed from group to group, segregation could be studied essentially independently of sintering by basing the studies on the average properties of the groups. Thus, as already detailed, the parts selection was composed of six groups, each consisting of eight parts made in sequence. The variance component associated with the differences between groups was termed the ‘macrosegregation variance’.

Apart from the necessity to randomize the parts in conjunction with the segregation component, the arrangement for sintering was otherwise influenced by an interest to examine three different aspects of sintering. It was this interest which led to dividing the sintering variance into three different components as mentioned earlier.

One of the indicated sintering aspects was concerned with the properties of parts pressed back to back and sintered side by side. To estimate the associated variance, it was necessary to keep such pairs of parts together during sintering. Thus, the parts arrangement for sintering was developed around pairs rather than individual parts. Since the effects of pressing, sintering and segregation would be expected to be minimal for such pairs, the associated variance was termed the ‘minimum process variance’. The second sintering aspect of interest was concerned with the differences within trays. This arose, in part, as a natural consequence of the necessity to randomize the parts in the trays. Since the randomizing procedure was of necessity implemented by pairs, the associated variance component was developed between pairs. It was termed the ‘sintering within trays’ component. Finally, the third aspect of interest had to do with sintering differences between trays. To examine this aspect, it was necessary to distribute the parts evenly between the trays and as indicated earlier, this was accomplished by using four parts or two pairs in sequence to represent each group in each tray. The associated variance component was called the 'sintering tray to tray' component.

To deal with all of the six parts properties of interest, it was actually necessary to conduct three different, albeit analogous, ANOVA studies. These are briefly indicated below in terms of the relevant variance components.

Crush Strength

The crush strength analysis involved four components as follows:

1) Combined Testing and Minimum Process Variance
2) Sintering Within Trays
3) Sintering Tray to Tray
4) Macrosegregation

The testing and minimum process components could not be separated in this case because as indicated earlier the crush strength test is destructive.

% Dimensional Change, Density and Hardness

The analyses of these properties involved five components as follows:

1) Testing Variance
2) Minimum Process Variance
3) Sintering Within Trays
4) Sintering Tray to Tray
5) Macrosegregation

The testing component in this instance included within part variations as well as measurement error.

Phosphorus Content

In this case the analysis involved six components as follows:

1) Testing
2) Microsegregation, (i.e. within part variations)
3) Minimum Macrosegregation Variance
4) Segregation Between Pairs
5) Segregation Within Groups
6) Segregation Group to Group

The terminology in this case is necessarily different than in the preceding analyses because sintering, of course, cannot effect phosphorus content on a part to part basis. However, other than terminology and the added step of separating within part variations as a pure component, the present analysis was essentially the same as the preceding analyses. In particular, components 3 through 6 respectively refer to the same part pairings and/or groups as components 2 through 5 of the % Dimensional Change, Density and Hardness analysis and as components 1 through 4 of the Crush Strength analysis.

PARTS TESTING PROCEDURE

% Dimensional Change and Density

The dimensional change characteristics and densities of the parts selected for the primary trial were determined in both the green and sintered conditions. The dimensional change values were calculated in percent versus the die. The densities were estimated on the basis of the part weights and the dimensional measurements as indicated below. The green properties were measured once and in accordance with the requirements of the ANOVA studies, the sintered properties were measured twice.

The inside diameter values were measured at the approximate mid height of the parts as the average of the high and low readings about the diameter using a bore gauge set against a flat cylindrical insert of standard height. The outside diameter values were similarly measured at the approximate mid height of the parts as the average of the high and low readings about the diameter using a bench comparator in combination with a V block. The comparator was set up prior to each set of measurements using a specially made stainless steel bushing machined to tight tolerances. The part heights were also measured using the comparator. In this case, the comparator was set up using a 2 inch gauge block. All of the dimensional measurements were made to the nearest 0.0001 inch. The part weight measurements were made to the nearest 0.01 gram using an electronic balance. The same balance was used for all measurements.

Mechanical Properties

The Rockwell B hardness of the parts was measured using standard equipment and techniques. Here again each part was tested twice. Both measurements were made in the top surfaces of the parts as pressed and sintered. Otherwise, the positions of the measurements were selected at random.

The crush strength of the parts was measured and calculated in accordance with the method of ASTM Standard B439-79.

Phosphorus Content

Following the crush strength tests, the parts were sampled for chemistry by drilling. Each part was sampled twice to a depth of about 1 inch through its top surface. The holes were approximately diametrically opposed and were otherwise positioned at random about the peripheries of the parts.

Atomic emission spectrometry was employed to analyze the samples. Briefly, the method consisted of dissolving a sample in an oxidizing medium and quantifying it as to phosphorus content versus a similar solution of a known standard.

Statistical Evaluations

The statistical evaluations were computer assisted using a SAS® software package.(13).

The indications of the data as to the overall or grand statistical properties of the parts of each of the mixes were evaluated. This included determinations of the mean, standard deviation and variance estimates of all of the parts properties involved. The determinations were carried out on a measurement to measurement basis so that the corresponding sample size was in most cases 96. Exceptions to this included the crush strength and phosphorus content for which the sample sizes were 48 and 192 respectively. The associated frequency distributions were evaluated for skewness and kurtosis and subsequently tested for normality. These determinations were conducted in accordance with the SAS software.

Statistical decision criteria were used in comparing the properties of the two mixes. In particular, indications of differences which were not found to be statistically significant were not considered to be physically significant. Mean values were submitted to a one-sided t-test and variances to a one-sided F test.(14) Both tests were conducted at the 95% confidence level.

The indicated ANOVA analyses were carried out in accordance with ANOVA theory. Here again, variance components which were not found to be statistically significant at the 95% confidence level were not considered to be physically significant.

Finally, there was interest in one instance to correlate two of the parts properties with each other and this was accomplished using simple linear regression techniques. The statistical significance of the resulting regression equation was checked in terms of its correlation coefficient using established methods.(15)

RESULTS AND DISCUSSION

Preliminary QC Results

The results of the preliminary Q.C. tests on the two mixes are shown in Table I.

Table I - Green and Sintered Properties of the Subject Mixes

Property/Component	Regular Mix	Binder Treated Mix
GREEN PROPERTIES		
Apparent Density (g/cm^3)	3.20	3.19
Hall Flow (sec/SOg)	31.5	29.0
Green Density @ 30 tsi, (g/cm^3)	6.75	6.75
Green Expansion @ 30 tsi (%)	0.13	0.15
Green Strength @ 30 tsi (psi)	1800	1790
Phosphorus Dusting Resistance	43.0	94.0
SINTERED PROPERTIES & CHEMISTRY		
Green Density (g/cm^3)		
Green Expansion (%)	6.80	6.80
Sintered Density (g/cm^3)	0.16	0.16
Dimensional Change vs. Die (%)	6.81	6.81
TRS (ksi)	-0.17	-0.18
Hardness (Rb)	114.2	111.2
Carbon (%)	50.0	51.0
Phosphorus (%)	0.026	0.025
Oxygen (%)	0.46	0.46
	0.57	0.061

A review of the results in Table I will show that apart from notable differences in flow, phosphorus dusting resistance and sintered strength, the two mixes were remarkably similar to each other in properties. The flow and dusting resistance differences which both favored the binder treated mix were expected; the sintered strength differences which favored the regular mix was not. Each of the three differences is briefly discussed below.

Flow Improvements

The 2.5 second improvement in the flow of the binder treated mix over the regular mix is a typical outcome of the differences in processing. In fact, much greater improvements in the neighborhood of 4 to 6 seconds are frequently observed. The basis of the improvements is thought to be a combination of the binder effect in agglomerating the fines and the fact that the binder is an essentially tact free solid at ambient temperatures.

Dusting Resistance Improvements

The improvement in phosphorus dusting resistance in the data was, of course, a natural consequence of the fact that the binder treatment was specifically designed to produce such an improvement. Thus, the important question in connection with this finding was not its origin but rather its meaning.

Interestingly, there were two different aspects to be considered in this regard: one had to do with the effectiveness of the binder treatment processing; and, the other, with the relative potentials of the two mixes.

The indications of the findings as to processing were that the binder treatment in this case had been about average. For reasons which have yet to be determined, ferrophosphorus seems to be one of the easier admix ingredients to bond. Consequently, phosphorus dusting resistance values in the mid-nineties such as the present value are rather the rule than the exception.

In contrast, since this study was one of the first to examine the effects of the binder treatment technology as practiced in this laboratory, there was very little applicable experience at the time to decide the potential of the mixes in terms of their dusting properties.

Earlier studies of the dust resistance test had shown that its mechanism involved intraparticle migration as well as dusting. More specifically it was found that susceptible particles in the test move through the powder sample under the influence of the elutriating gas until they reach the surface and only then do they escape by dusting. Thus, the present results were very much an indication of segregation resistance as well as of dusting resistance. However, beyond this, their meaning was essentially uncertain. In particular, it was not known whether the dusting resistance value of the binder treated mix was sufficiently greater than that of the regular mix to effect parts variability improvements or not.

An interesting aspect of this situation was that virtually all of the work done on the binder treatment method in the laboratory to this point was based on the tacit assumption that dust resistance improvements of the indicated magnitude would be sufficient to improve variability performance. Thus, in addition to being a means of assessing the potential of the method, the present study was also a test of an important underlying assumption.

Of course, support for the indicated assumption already existed in the form of earlier reported studies along similar lines by researchers at Hoganas AB in Sweden.(16) However, while these reports were helpful, they were not directly applicable. The

Swedish studies were based on a different process, different binders and a different method of testing for dust resistance.(17)

Sintered Strength Difference

The sintered strength difference in the preliminary data was a matter of some concern. The difference was small but based on all of the work which preceded these studies as well as all of the precautions which went into conducting them, it should not have been there. Unfortunately, due to scheduling constraints connected with the availability of the pressing facilities, there was no time to make the mixes over or else this option would have been exercised. Nor did later investigation of the effect lead to an explanation.

PRIMARY STUDY RESULTS

The grand statistical results of the study are presented in Table II below. The first four columns of the table list the means and standard deviation values of the six parts properties of each mix. The standard deviation values are presented instead of the corresponding variance values because they have the same units of measurement as the associated mean values and are thus, more readily understandable. The fifth column of the table lists the answer to the question: Is the variability estimate of the regular mix, i.e. S^2_R statistically significantly larger than that of the binder treated mix, i.e S^2_B. A YES answer indicates that the data were conclusive in this regard at the 95% confidence level. A NO answer, on the other hand, simply means that the data were inconclusive. The sixth column of the table indicates the percentage improvement of the binder treated mix over the regular mix in terms of the corresponding standard deviation values for those cases where the variance differences between the two were found to be statistically significant.

Table II - Grand Statistical Results

Property	Binder Treated Mix		Regular Mix		Is $S^2_R > S^2_B$?	% $\frac{(S_R - S_B)}{S_R}$
	Mean	Std. Dev.	Mean	Std. Dev.		
Sint. Dens. (g/cm^3)	6.809	0.0116	6.831	0.0124	NO	--
I.D. Dim. Chg. (%)	-0.2489	0.0167	-0.2587	0.0271	YES	38.4
O.D. Dim. Chg. (%)	-0.2334	0.0176	-0.2546	0.0255	YES	31.0
Hardness (Rb)	47.0	1.16	49.6	1.50	YES	22.7
Crush Str. (ksi)	92.2	1.28	95.5	2.06	YES	37.9
Phosphorus (%)	0.462	0.010	0.452	0.016	YES	37.5

A review of the data in Table II will show that the variability of the regular mix exceeded that of the binder treated mix in five of the six properties of interest. The exceptional property was sintered density but even in this case, the relative values of the two favored the binder treated mix. In terms of the standard deviation values, the greatest improvement in the binder treated mix was in the I.D. Dimensional Change property and the least improvement was in the Rockwell hardness. The corresponding values were 38.4 and 22.7% respectively. The general improvement in the binder treated mix as averaged over all five properties was 33.5%. Evidently, the assumption underlying the development of the binder treatment method on the basis of improved dusting resistance results was a reasonably good one.

A closer inspection of the data in Table II will show that the two mixes also differed in their mean values in all six properties. In many cases, the differences are numerically small but all are statistically significant at the 95% confidence level.

Of these differences, the crush strength difference is perhaps the most important from a practical standpoint. In this case, the performance of the binder treated mix was relatively unfavorable in that its value fell short of that of the regular mix by upwards of 3000 psi.

Interestingly, there were two possible explanations of this result. One was that it had the same cause as the sintered strength difference indicated in the preliminary test results. Another was that it was due to the fact that in the primary trial the binder treated mix had a slightly lower sintered density than the regular mix.

The latter explanation was suggested by a special analysis which showed the existence of an extremely strong correlation between crush strength and sintered density in the regular mix data. This particular result was as follows:

$$CS_R \quad 177.2\, D_R - 1{,}115.0;$$

where CS_R and D_R represent the corresponding regular mix crush strength and sintered density values respectively. The associated correlation coefficient was 0.976 and the equation was significant at the 99% confidence level.

The idea that the lower crush strength value of the binder treated mix was due to its lower sintered density value was based on the indications of extrapolations of this equation. In particular, when the crush strength according to the equation is evaluated as a function of the sintered density mean of the binder mix, the resulting value is within a few hundred psi of the value which was actually observed, (e.g. 91.6 versus 92.2 ksi).

The reason that the binder treated mix had a lower density than the regular mix in the trial was due in part to the greater weight loss associated with the binder addition, (0.O1 g/cm^3), and in part to the fact that the mix was actually pressed to a slightly lower density, (also 0.01 g/cm^3).

Analysis of Variance Results

The implications of the data in Table II as to the cause of the improved variability performance of the binder treated mix are that it is an effect of the associated reduction in the phosphorus variance of the mix. As will be seen, the ANOVA results generally support this view but go beyond to show that the segregational differences were predominantly in microsegregation rather than in macrosegregation.

Of the six ANOVA studies which were conducted, all were reasonably consistent in regard to their physical and metallurgical indications. However, the studies of sintered density and hardness were relatively weak from the statistical standpoint. Several components which were strongly indicated in the other studies were only weakly indicated in these studies. To be precise, the associated confidence estimates fell below the 95% level. Consequently, the results were not considered physically significant and are not reported.

The results of the studies of the other four properties are presented in Table III. To facilitate interpretation of the data, the variance components of both mixes are indicated in common terms, i.e. - as fractions of the corresponding overall regular mix variance in each case. Only those components which were statistically significant at the 95% confidence level or higher are reported.

Table III Analysis of Variance Results Per Unit of Total Regular Mix Variance

Property	Component	Binder Treated Mix	Regular Mix
% I.D. DIMENSIONAL CHANGE	Testing Minimum Process Sintering Within Trays Macrosegregation	0.041 0.101 0.222 nil	0.026 0.203 0.487 0.284
%O.D. DIMENSIONAL CHANGE	Testing Minimum Process Sintering Within Trays Macrosegregation	0.018 0.091 0.282 nil	0.032 0.085 0.587 0.296
CRUSH	Combined Testing &		

STRENGTH	Minimum Process Sintering Within Trays Macrosegregation	0.321 nil 0.126	0.308 0.197 0.495
% PHOSPHORUS CONTENT	Testing Macrosegregation Within Group Segregation	0.123 nil 0.104	0.249 0.393 0.245

An analysis of the data in Table III showed that the dimensional change and crush strength studies had several points in common. Consequently, it will simplify matters somewhat to treat the results of these studies jointly.

The indicated points of commonality in the three studies were also the principal findings of the studies. They were as follows:

1) Sintering differences tray to tray did not contribute significantly to the variances of either mix.

2) The remaining sintering sources including the minimum process and within tray sources generally contributed to the variances of both mixes; the important difference being that they added substantially more to the regular mix than to the binder treated mix.

3) In addition to the sintering sources, the macrosegregation or group to group sources also contributed substantially to the regular mix but little or nothing to the binder treated mix.

The remaining ANOVA results in Table III are those of the phosphorus content study. These findings indicated the presence of significant microsegregation in the regular mix but not in the binder treated mix. In addition, there were also indications in the data of the presence of macrosegreqation which took the form of within group variations. In this case, the indications were that both mixes were affected with the regular mix being the more affected of the two.

A general analysis of the findings of the ANOVA studies led to the view that the microsegregation which was indicated in the results was the basic underlying cause of the differences in the variability performance of the two mixes. This view arose in part because the microsegregation difference in the mixes was the largest known significant difference and in part because microsegregation as a cause provided a satisfactory explanation of the other findings of the study.

In contrast, the within group indications of macrosegregation in the ANOVA data were not thought to be very important at all. These findings did not correlate with any of the findings of the other ANOVA studies. In addition, and perhaps more significantly, the associated differences in phosphorus variation which they indicated were, at best, rather small.

The interrelation of the microsegregation finding to the other findings of the ANOVA studies was as follows. Based on the results of the dimensional change and crush strength studies, the relatively poor performance of the regular mix in comparison with the binder treated mix could be characterized as the result of two differences. One was that the regular mix was more sensitive to process variations in sintering. The other was the presence in the regular mix of unfavorable variations on a group to group basis. Both of these differences could be explained as effects of microsegregation.

Of the two, the idea that microsegregation or within part variations should result in increased sensitivity to external variations whether they be in sintering or some other process step is thought to be both reasonable and fairly obvious. In contrast, the idea that microsegregation could be connected with unfavorable variances on a group to group basis may need explanation. In the context of the study, significant compositional variations group to group would be macrosegregation not microsegregation. However, if it is considered that macrosegregation is not limited simply to gross variations in average composition, then the implied contradiction is only an apparent one. In particular, macrosegregation in its most general form may also be manifest as significant changes in the pattern of microsegregation and this is precisely what is thought to have occurred in the present case. More specifically, it is thought that the explanation of the group to group variations of the regular mix was that the microsegregation changed group to group while remaining reasonably the same among parts within a group.

POTENTIAL BENEFITS OF BINDER TREATED MIXES

The original objective of the research which led to the binder treatment method was to develop premixes which would be more economical to process than existing premixes. It appeared that there were two ways in which to do this. One was to improve flow and the other was to improve premix uniformity with respect to alloy content. As initially conceived, these improvements were seen as possibly conflicting and it wasn’t until much later that it was realized that both could be achieved simultaneously. Without going into detail, all that was necessary was to abandon the traditional wisdom in connection with binders and binder treatment.(18)

In any case, the aim in improving flow, of course, was to permit increased pressing rates. The aim is improving premix uniformity was precisely as has been shown to reduce the variability of the resultant parts.

The indicated variability reductions were seen as potentially offering several different opportunities for economic advantage. These opportunities are perhaps most apparent when the reductions are considered in terms of the now familiar concept of Statistical Process Control.(19)

Figure 5 presents a schematic of a parts making control chart. For purposes of discussion, the control lines are indicated to be the 3 Ó limits of a parts property as determined from experience in producing parts from a regular mix. The engineering or customer specification requirements of the property are also shown in the diagram. These are indicated as slightly bracketing the control limits.

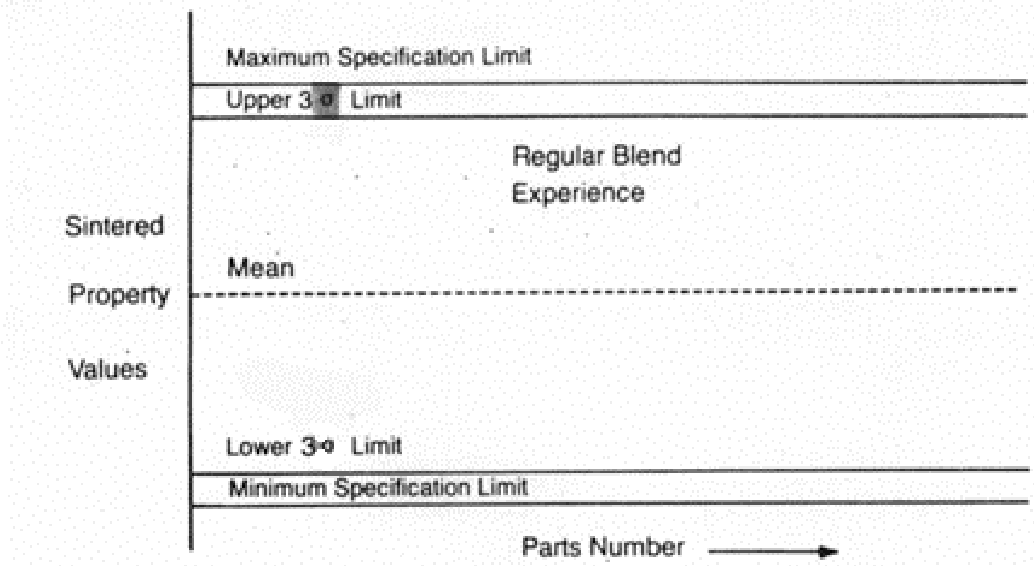

Figure 5 Control Chart Schematic of a Regular Blend Property

In comparison, Figure 6 presents a similar schematic which additionally incorporates the results of a binder treated mix. For the sake of realism, the positions of the various lines in the diagram were determined in accordance with the results of the present findings as indicated in Table II The particular property used for the purpose was the % I.D. Dimensional Change.

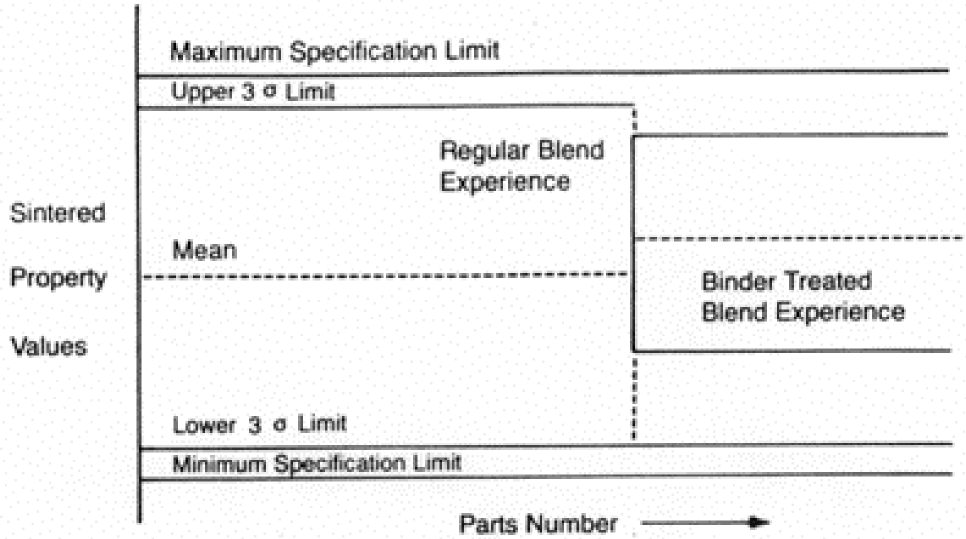

Figure 6 Control Chart Schematic Comparing Regular and Binder Treated Blends

The changes occasioned by the transition to the binder treated mix in Figure 6 include a tightening of the control limits and a slight displacement of the chart mean. This latter change may be amendable through minor modifications in processing or by slightly altering the raw materials of the mix. On the other hand, it may simply be a characteristic of the technology. This is an aspect which remains to be seen.

In any case, in terms of the concepts of Statistical Process Control, the changes indicated in connection with the binder treated mix represent a quality improvement. In fact, as those familiar with this particular method of parts making control will agree, the magnitude of the changes shown represent a very substantial quality improvement.

The economic benefits which may be derived from such an improvement will depend on the specific part application involved. If the application is such that the customer can make use of the associated reduction in property variability, then the economic advantage to the parts maker is increased competitiveness. This is possibly the most important potential advantage of the binder treatment technology.

If the part application is such that the variability reduction cannot be used because the engineering requirements are already as tight as they need be for optimum performance, then it should be possible to derive advantage from the improvement in the form of reduced costs. There are three general areas of opportunity as follows:

1) Reduced testing requirements;

2) Reduced rejections due to fewer out of control incidents; and,

3) Increased sintering productivity.

Each is briefly discussed below.

Reduced Testing

Design of a Statistical Process Control system is often based on the results of a preliminary ANOVA study and includes an analysis to decide the required sample and test per sample frequencies needed to implement the method. Very often this analysis includes consideration of test costs and results in a formal calculation procedure aimed at devising a scheme which both satisfies the system requirements and minimizes the cost. Ordinarily, while several factors may be involved in this calculation, some value indicative of the variability of the parts or a parameter directly related thereto will be one of the major factors. In general, the relation is one in which the test requirements increase as the variability value increases. Thus, a significant reduction in parts variability such as that in the present case would represent a valid reason to review the testing requirements and may result in a substantive, albeit modest, decrease in costs.

Reduced Rejections

Substitution of a binder treated mix for a regular mix without compensatory changes in the engineering requirements of the parts is essentially the situation depicted in Figure 6. The resulting increase in the difference between the requirements and the limits of variability of the new mix which is shown in the figure basically represents increased latitude for error elsewhere in the process. In other words, according to the figure, the probability that a temporary loss of control will result in a rejection has decreased. Consequently, the number of rejections due to such incidents may reasonably be expected to decrease.

In addition, there should also be fewer rejections as a consequence of the inability to react appropriately to gradual progressive changes indicating a chronic out of control situation. In particular, the increased latitude mentioned above would be expected to provide increased time to locate the source of a problem and implement the necessary remedial measures before the situation deteriorates sufficiently to cause significant numbers of bad parts.

Increased Sintering Productivity

The suggestion that the use of binder treated mixes has a potential for economic advantage through increased sintering productivity differs from each of the foregoing suggestions in that it is more a matter of speculation than reasonable expectation. In addition, as visualized, the advantage could only be realized through major changes in the sintering process and thus would require considerably more input from the parts maker than either of the earlier suggestions. However, the idea is possibly a good deal more interesting too because even a rather small productivity increase and consequent costs savings in the sintering step would be expected to have a very substantial effect on profitability.

The technical basis of the idea is the fact that variability as employed in Statistical Process Control is a systems property and not simply a mix property. In sintering, the system includes the sintering conditions and equipment as well as the mix. Therefore, it may be possible to compensate the variability reduction associated with a change in the mix by changes in sintering. The sintering changes visualized would include increases in furnace loading and/or belt speed and any other changes within the limits of practicality that are necessary to effect the conditions of control which existed prior to the change in the mix. Minor changes in the mix itself would probably be helpful in these regards and might actually be necessary. Of course, here again, any changes that would be made would be subject to practical consideration.

SUMMARY OF FINDINGS AND GENERAL CONCLUSIONS

The original objective of the study was to estimate the potential of a binder treatment method as developed in Hoeganaes Laboratories in the last several years. In general, the results of the study showed that as applied to an iron powder mix containing 0.45% P in the form of ferrophosphorus, the method effected significant improvements in powder flow, phosphorus dusting resistance and most importantly in the variability performance of parts made from the mix. The flow rate improvement was considered to be an effect of the treatment in agglomerating the very fine particles of the mix. The phosphorus dusting resistance and variability improvements were regarded as being correlated and both were considered to be due specifically to the bonding of the ferrophosphorus to the iron.

In the parts making portion of the study, variability performance was examined in terms of six properties. Compared to a regular mix, similarly made parts of the binder treated mix exhibited statistically significant variability improvements in five of the six. The average improvement in these five in terms of the corresponding standard deviation values was 33%.

The parts making study also showed the existence of statistically significant mean value differences between the two mixes. In this case, the differences were in all six properties. However, they were also all small and, therefore, questionable as to physical significance. For example, the most significant difference which happened to be in crush strength was less than 4%. It was speculated that this difference as well as the other differences may be amendable via minor modifications in processing and/or premix materials.

An analysis of variance study was incorporated into the parts making trial in an effort to get a better understanding of the findings. The study was designed specifically to assess the contributions of sintering and segregation. The results showed that both were effective in contributing to the higher variability of the regular mix. Interpretation of the findings led to the view that of the two, segregation was the more important. The data suggested that it was the principal underlying cause of the observed sintering effects. The presence of microsegregation was clearly shown for the regular mix and the presence of macrosegregation was indicated. Both were essentially absent in the binder treated mix. The macrosegregation referred to was speculated to be in the form of a change in the pattern of microsegregation in parts representing widely divergent portions of the mix.

Finally, the potential economic advantages to be derived from the use of binder treated mixes were discussed. It was suggested that the associated flow rate improvements are applicable to increase press rates. In the case of the variability improvements, it was pointed out that the advantages will depend on the particular part application involved. If the application is such that the variability reduction can be realized as a true quality improvement, then the advantage is increased competitiveness. If on the other hand, the application is such that there is no benefit to be derived in terms of quality to the customer, then the variability reductions have application to reduce costs. Three possibilities in this connection were cited including: reduced testing; reduced rejections; and increased productivity in sintering. Each of the three was discussed briefly to indicate how a cost savings could be effected.

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