

Effect of Process Variables on the Dimensional Change of Atomised Ferrous Powder Sintered in Nitrogen Atmosphere

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Abstract - Powder metallurgy is a net shape manufacturing approach, which is very attractive because of its ability to form powders directly into finished components. These components are formed in an economical manner having a considerable choice in material properties, mechanical, chemical and microstructure control. The chemical composition of powder blends and their consolidation are characterized producing a full fine pearlitic structure during the sintering process which is important to maintain tensile strength and size control. Changes in dimensions of metal powder compacts do occur during the production process. The ability to control the changes is an important part of the manufacturing process. The outstanding savings achieved through the production of near net shape is the driving force behind this manufacturing process. Size change of the material must be known and it is one of the most important functions when designing compaction dies for new components. In this paper, an overview of the effect of Dimensional change (Growth) during sintering when compared to the two atomized pre alloyed grades of ferrous powders was investigated. Density, spring back, dimensional change were determined in the green and sintered conditions. Densities in the range of 6.0 – 6.6 g/cm³ were achieved by withdrawal die motion and sintering temperature of 1100 °C, 1110 °C and at 1120 °C were utilized. Sintering temperature, green density and % of carbon content were found to be the most significant variable controlling dimensional change.

Keywords - Dimensional Change, Fe-C-Cu parts, Powder Metallurgy, Sintering temperature.

I. INTRODUCTION

The sintering of iron – copper – graphite mixtures involves, solid bonding of iron to iron, diffusion of carbon into iron, melting of copper, solution and diffusion of copper into the iron grain boundaries. Diffusion of carbon is always completed before the copper attains its melting point. Carbon has no effect on the solubility of copper at the iron grain boundaries, but the rate of solution is decreased. Changes in dimensions of metal powder compacts do occur during the manufacturing stages. The capability to control the changes is an important part of the production process. The remarkable savings achieved through the manufacturing of near net shape is the motive force behind this production process. Dimensional change

plays a vital role in PM component manufacture. Managing the dimensional change occurring in the sintering process in order to meet the required part dimension is essential. Powder properties and the process parameters both influence dimensional change. Powder properties that influence dimensional change include change in compact density, change in carbon addition, change in copper addition, increase or decrease in hydrogen loss, insufficient iron oxide reduction, change in sintering time, variable cooling rate after sintering, parts decarburized, parts excessively carburized and parts oxidized.

Specific surface area depends on the shape of the powder. A powder with highly irregular particle shape will have a larger specific surface area compared to the one which is nearly spherical in shape. Particle size is not an easily quantifiable powder property. Comparison of particle size of two or more powders that have similar particle size distribution can be done by apparent density and flow rate. If two powder lots have very similar particle size distribution but have different apparent densities, then the one having the lower apparent density would be considered to be the more irregularly shaped powder. Particle size is responsible for the material to be compacted to a range of densities. Particle shape determines the interlocking and interaction of the powder grains during compaction. Particle size distribution contains consistent flow and apparent density. Particle density influences the porosity level of the component.

Particle hardness and its chemical composition has a direct effect on the compaction force required to achieve the set out density specification of the component. Other factors that influence the rate of sintering, and hence can have an effect on the dimensional change are lubricant and other additives present in the powder.

Process variables that influence dimensional change include Apparent density, Flow rate, green density, chemical composition, particle size distribution, nature of lubricant, amount of lubricant, press tool design and tolerances, tool alignment, compacting pressure, speed of press, dew point of the sintering furnace, time, temperature, phase transformation, rate of heating, rate of cooling, and type of atmosphere. All sintering related parameters cannot be easily quantified as in the case of primary briquetting related parameters namely the green density and briquetting pressure.

The dew point of the sintering furnace, rate of heating, rate of cooling and time and temperature are the sintering variables that are more difficult to measure. The two major process variables that influence the degree of sintering are

sintering temperature and time. To achieve the desired dimensional change and sintered density control of these two variables, in addition to that of the green density, chemical composition is essential.

The greatest challenge faced by the parts maker is the close control of dimensional change, combined with the requirement of a high sintered density. The percentage of carbon additions affect the dimensional change of iron powders. Shrinkage is higher at higher carbon levels. Copper, which is added for strength purposes, increases growth. The size change is generally defined as a diametric difference between the compact to sinter or between the die size and the size of the sintered component at room temperature expressed as a percentage. The die to sinter is more frequently used in the powder metallurgy industry for practical reasons. The green to sinter dimensional change is used for calculating the growth and shrinkage during sintering process.

The importance of this study is to determine the effects of powders and process variables on the dimensional change of atomized grades of ferrous powder. The factors included in the study are grades of ferrous powders, apparent density, flow ability, amount of lubricant, carbon %, Copper %, density of compact, and the sintering temperature. These eight factors not only have a significant effect on dimensional change, but are also the ones that are more repeatedly modified.

II. PREVIOUS RESEARCH

[13] have investigated dimensional change on stainless steel powder and the results show that temperature and fines content of the powder have a strong influence on the dimensional change. [7] have investigated improvements in dimensional stability in starmix powder can be attained by controlling the segregation of the powder mix and die filling. [9] have studied that close dimensional stability can be controlled by bonding of powder mixes that influences the flow behavior. [6] compared dimensional capability using Cr-alloyed materials verses conventional P/M alloyed. [12] has investigated the dimensional consistency in AHC 100.29 base powder can be achieved by controlling the dimensional tolerance, composition of the mix, segregation in powder, pressing and sintering. [14] have briefed a relationship between microstructure and dimensional change in selecting the appropriate alloying system for the desired performance and tolerance level in Astaloy powder. [8] have developed a model by applying a multilayer neural network using back propagation algorithm. [11] have investigated in Astaloy CrL powder containing Cr, Mo and Mn about the influence of sintering temperature and component density on the mechanical properties and found higher temperature produced increased tensile strength. [10] have investigated in Astaloy CrM powder containing Cr about the influence of sintering parameter on the mechanical performance. [15] have studied the consolidation of iron powder that shows impact on lubrication procedure, Compacting pressure and sintering temperature. [5] have studied the

effect of increase in carbon content and master alloy concentration leads to the increase in steel strength. [16] have studied the effect of carbon, sintering temperature, and cooling rate had marked effect on physical and mechanical properties of Fe – 1.5 Mo Astaloy powder.

III. MATERIALS

The metal powder used in this work was the atomised iron powder grades manufactured by Hoganas India Private Limited. The atomized iron powders used in the study have good compressibility, high chemical purity, good alloying properties, low H₂ loss, good grain size distribution, good grain shape, good internal particle friction and good internal particle structure. The copper powder having good chemical purity, good grain size and grain shape is spherical. The carbon powder is 99.99 % pure with low ash content.

The chemical composition is a significant factor in determining powder reactions to conditions of initial compaction, specifically for the powder to densify. During sintering many chemical reactions are possible between the admixed alloys and the sintering temperature. Subsequently, the properties of the final sintered material greatly depends on the composition of the material. Table 1 lists the chemical composition of the atomized iron powders.

Table 1. Chemical Compositions of Atomised Iron Powders

Mix	Premix	Type	Iron	Copper	Carbon	Lubricant
1	ASM 25	Atomised	Base	2.0%	0.2%	0.8%
2	ASM 25	Atomised	Base	2.0%	0.8%	1.0%

The study utilized two atomized iron powders that differed each other in % of carbon and % of lubricant. Table 2 lists the particle size distribution, apparent density (AD) and the flow rate of the atomized iron powder.

Table 2. Particle size distributions, AD, and Flow rate of the Atomised Iron powders

Mesh fraction (Micron)	ASM 100.25	ASM 100.25
+ 250	0.0 max %	0.0 max %
- 250 +150	15.0 max %	15.0 max %
- 150 + 63	Bal	Bal
- 63	25.0 – 45.0 %	25.0 – 45.0 %
AD, g/cm ³	2.87	2.97
Flow rate, Sec /50g	30.5	34

IV. PROCESSING

Test specimens used in the study were in the form of powder compressibility test specimen having the dimensions specified in [1] - [2] (nominally 25.38 mm x 7.05 mm) using a Dorst 50 ton mechanical press. The bush used in the study is shown in Fig. 1.



Fig.1. Bush used in study

From each of the two powder samples, three sets of green compressibility specimens each having a different green densities were compacted. The three green densities were 6.0 (+/-0.05) g/cm³, 6.3 (+/-0.05) g/cm³, and 6.6 (+/-0.05) g/cm³. Three repetition specimens were used for each combination of powders. The dimensions, spring back, weight, volume and densities of the compacted specimens were determined. Green density was determined by water immersion as per [4]. Sintering was performed on a 606 mm (24 inch) Abbott mesh belt furnace. The bushes were sintered for 30 minutes in a 100 % nitrogen atmosphere. Specimens of each powder sample and green density were sintered in the furnace at the selected temperature. Three sintering temperatures were adopted : 1100 °C, 1110 °C, and 1120 °C. Sintered specimens were measured for determination of density and dimensional change. Sintered density was determined by water immersion as per [4]. Sintered dimensional change was determined as per [3].

V. RESULTS AND DISCUSSIONS

Table 3 shows the briquetting pressures used and the spring-back observed for all groups of powders. The elastic expansion of the compact after ejection from the compacting die is called spring-back and is measured according to the following Eq.(1) [17].

$$s (\%) = 100 (\lambda c - \lambda d) / \lambda d \quad (1)$$

Where $s (\%)$ = Spring -Back (%),

λc = Ejected compact transverse dimension,

λd = Compacting die dimension.

Table 3. Briquetting Pressures used and Spring-Back observed for the Compacting process studied

Powder Type	Powder Grade	Briquetting Pressure And Spring-Back	Green Density, g / cm ³		
			6.0	6.3	6.6
A	ASM 100.25	Briquetting Pressure, MPa	226	286	362
		Spring-Back, %	0.04348	0.05139	0.06325
B	ASM 100.25	Briquetting Pressure, MPa	213	273	355
		Spring-Back, %	0.04744	0.05139	0.07511

Table 4. shows the green density, sintered density, and dimensional change of all sample sets. All dimensional change determinations were based on the transverse dimension of the specimen (25.38mm, 7.05mm). Dimensional change was determined both as 'green-to-sinter' and 'die-to-sinter'. Data shown are the average values of dimensional change for each set of three samples. Dimensional change was calculated using the Eq. (2) and (3) listed below [13].

Dimensional change 'green-to-sinter' (%) = (Sintered outer dimension – Green outer dimension) ÷ (Green outer dimension) x 100 % (2)

Dimensional change 'die-to-sinter' (%) = (Sintered outer dimension – Die inner dimension) ÷ (Die inner dimension) x 100 % (3)

For all data analyses and correlation, only the dimensional change green to sinter data were used. Since, the green dimension equals die dimension + spring back, theoretically dimensional change die to sinter equals dimensional change green to sinter + (spring back ÷ green dimension). In some cases, sintering led to shrinkage of the specimen, hence resulting in negative values for dimensional change green to sinter and expansion of the specimen, hence resulting in positive values for dimensional change green to sinter. Two measurements were made 90° to one another as shown in Fig.2. The two measurements were then averaged to determine an overall dimensional change.

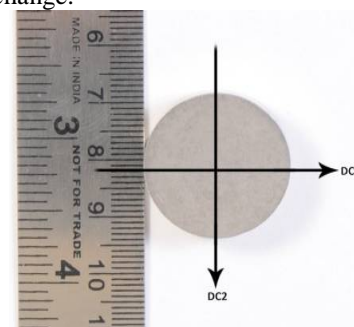


Fig.2. Direction of Measurement

The factor affecting the densification of green compacts from the metal powder is the green density of the specimen or the compacting pressure at which the specimen is pressed, which ascertain green density. The dimensional change obtained for atomized iron powder are shown in Fig.3 (a, b, and c) for the low, medium and high green densities respectively. The dimensional change of compacts from atomized iron powder is plotted as a function of the carbon content in Fig 4 (a, b, and c) for the low, medium and high green densities respectively. The change in carbon addition and amount of lubricant which shows a greater effect on dimensional change in atomized grades of iron powder is decreasing in the graphs. The plots of dimensional change versus green density for the low, and high carbon contents respectively in fig 5 (a, b, and c). The higher the density, the smaller the sintering expansion. The sintered density is much improved by the higher green density. Fig.6 (a, b, and c) through Fig.8 (a, b, and c) show the dependence of sintered density versus sintering temperature, carbon content and low, medium and high green densities of atomized iron powders respectively.

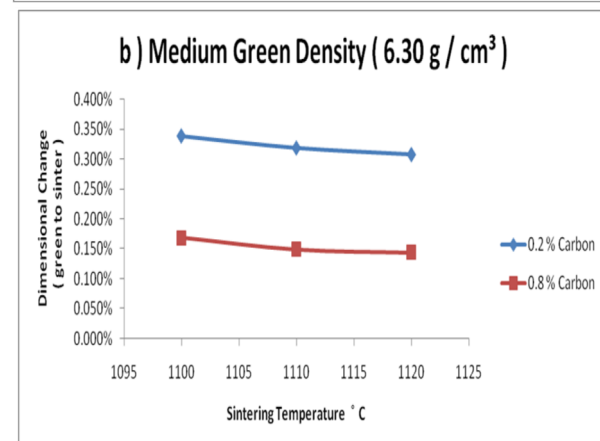
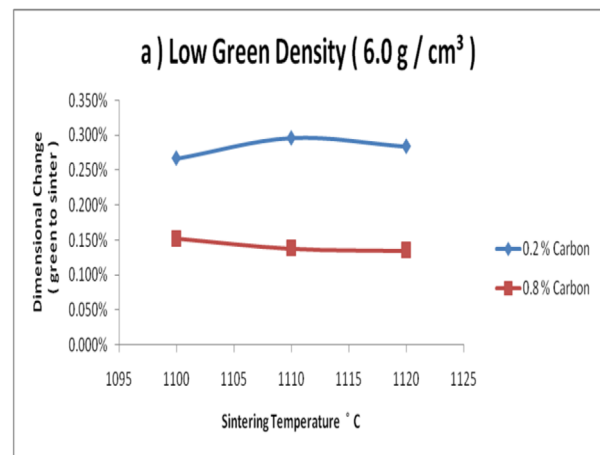
In Fig.3 (a, b, and c), the relationship between the dimensional change and sintering temperature is not exactly linear but the line does curve slightly downwards at the 1120° C temperature mark, indicating that at this higher temperature there was a more rapid reduction of surface oxides, copper migration to the grain boundaries and fusion of iron grains. Carbon diffuses readily into iron within five minutes at a temperature of 1040° C and give the carbon enough time to diffuse into the iron, leading to longer effective sintering duration. Similar effect was also reflected in the sintered density versus sintering temperature plots in Fig.6 (a, b, and c). Where the line curves upwards.

The effect of each of the three variables on the dimensional change was determined from the slopes of their respective plots in Figs. 3, 4, and 5. The slope of the line drawn through the 0.8 % carbon content data in Fig.3 (a) was used to represent the effect of sintering temperature on the dimensional change for the low green density components (which is found out to be 0.0085 % dimensional change (green to sinter) per 10 °C). Similar data were determined for the medium and high green density component groups having 0.8 % carbon content.

The effect of carbon content on the dimensional change, slopes of the plots in Fig.4 (a, b, and c) were obtained to represent low, medium and high green densities respectively. These findings were based on the 1110° C data only. The effect of green density on the dimensional change, slopes of the plots in Fig. 5 (a, b, and c) were obtained to represent low, medium and high green densities respectively. These findings were based on the 1110° C data only. Based on the above calculations, the response of dimensional change to a measurable change in each of the controlling parameters (Sintering temperature, carbon content, and green density) were determined as shown in Table 5.

Table 4. Summary of dimensional change, green density and sintered density data of Atomized ferrous powder for the Various process parameters studied

Part Density Group	Sintering Temp ° C	Powder Type	% C	Green Density g / cm ³	Sintered Density g / cm ³	Dimensional Change green to Sinter	Dimensional Change Die to Sinter
Low Density	1100	A	0.2	6.06	6.03	0.267 %	0.317 %
		B	0.8	6.04	6.13	0.152 %	0.199 %
	1110	A	0.2	6.06	6.13	0.296 %	0.347 %
		B	0.8	6.04	6.23	0.138 %	0.185 %
	1120	A	0.2	6.06	6.23	0.284 %	0.334 %
		B	0.8	6.04	6.28	0.135 %	0.181 %
Medium Density	1100	A	0.2	6.34	6.36	0.339 %	0.391 %
		B	0.8	6.29	6.45	0.168 %	0.220 %
	1110	A	0.2	6.34	6.38	0.319 %	0.371 %
		B	0.8	6.29	6.48	0.149 %	0.200 %
	1120	A	0.2	6.34	6.40	0.308 %	0.360 %
		B	0.8	6.29	6.50	0.144 %	0.195 %
High Density	1100	A	0.2	6.61	6.70	0.336 %	0.399 %
		B	0.8	6.72	6.76	0.183 %	0.256 %
	1110	A	0.2	6.61	6.72	0.312 %	0.375 %
		B	0.8	6.72	6.80	0.165 %	0.239 %
	1120	A	0.2	6.61	6.74	0.306 %	0.324 %
		B	0.8	6.72	6.83	0.179 %	0.252 %



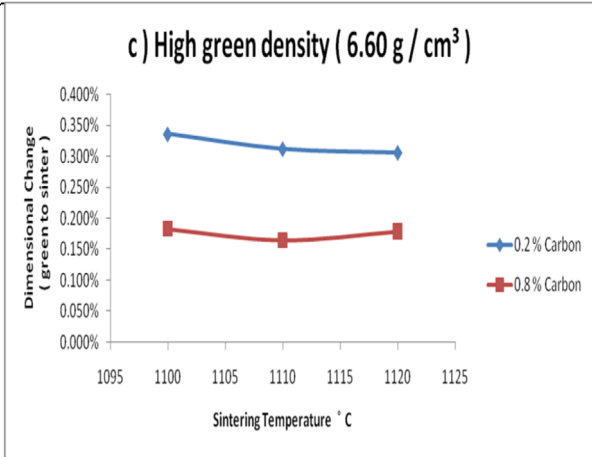


Fig.3 (a, b, and c) : Dimensional change versus the sintering temperature for three different green densities

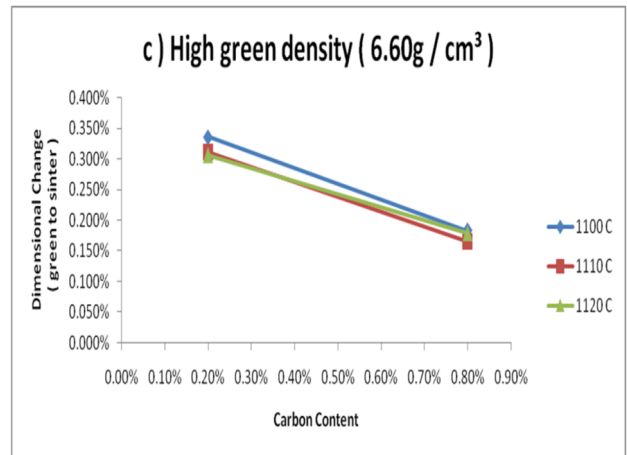


Fig.4. (a, b, and c) : Dimensional change versus the carbon content for three different green densities

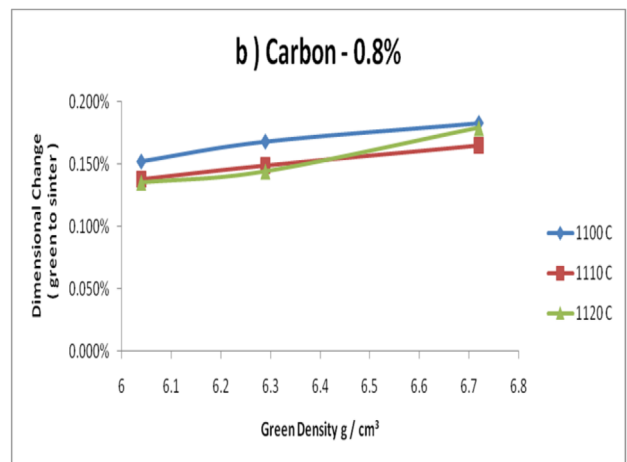
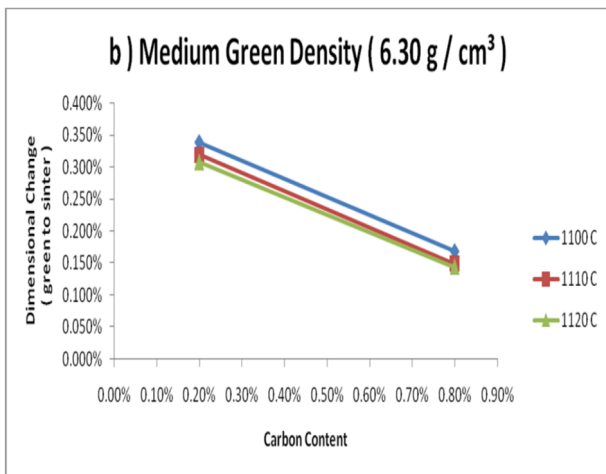
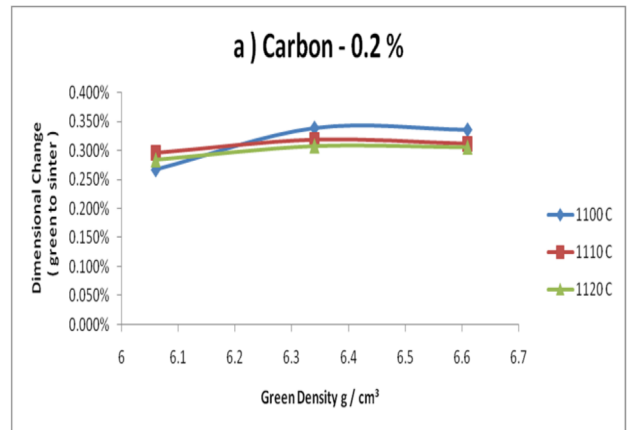
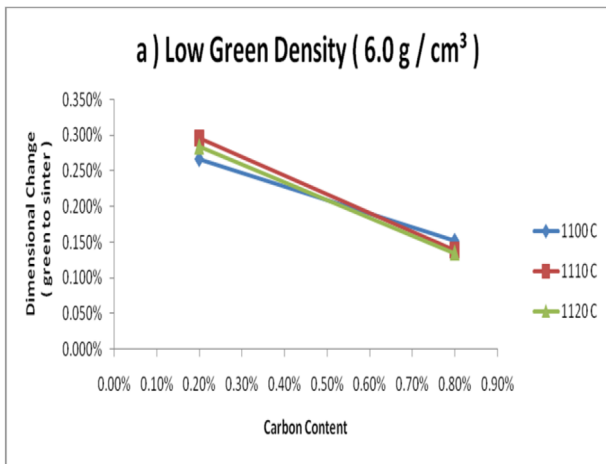


Fig.5. (a and b) : Dimensional change versus the green density for three different green densities

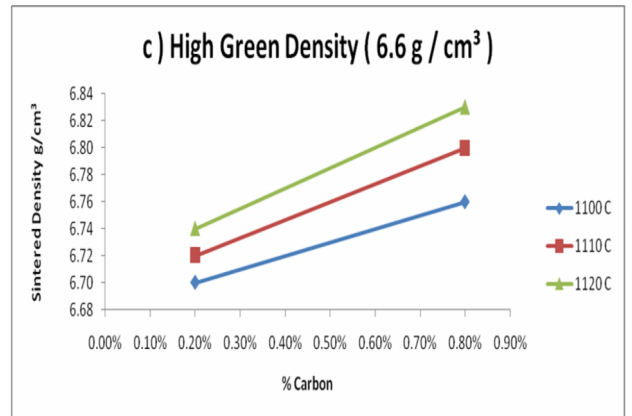
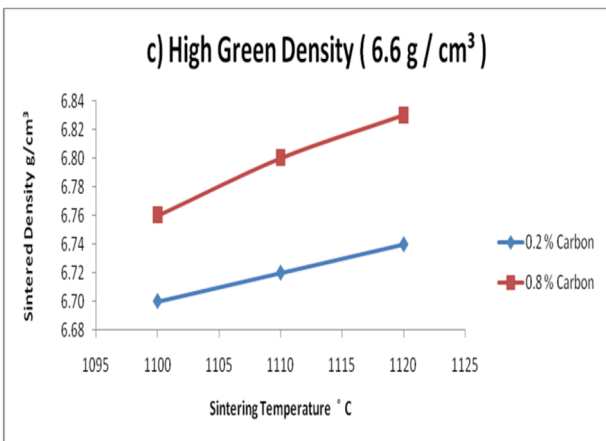
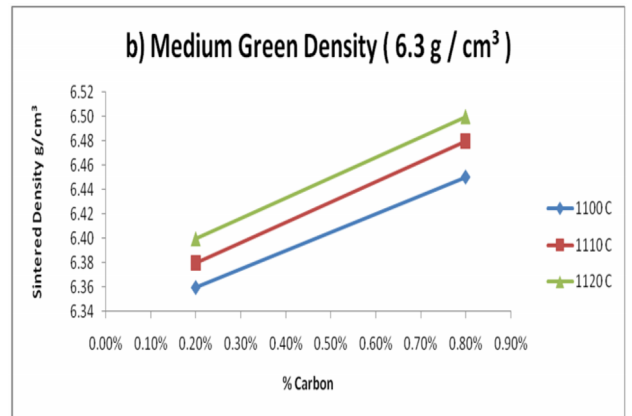
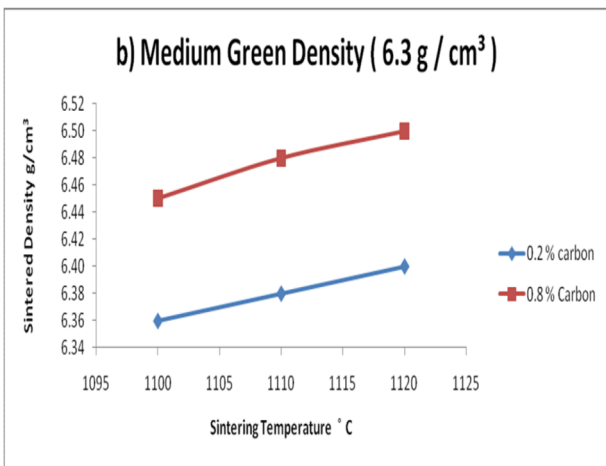
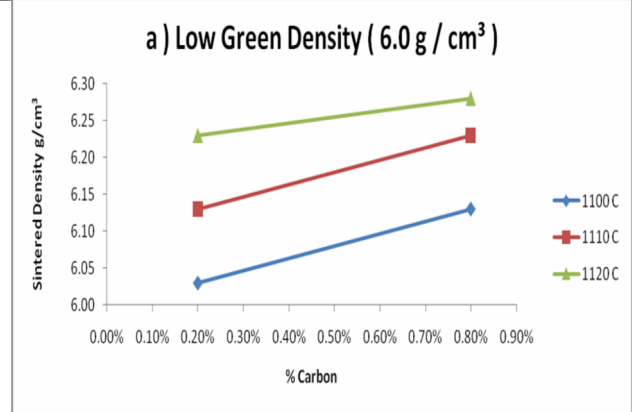
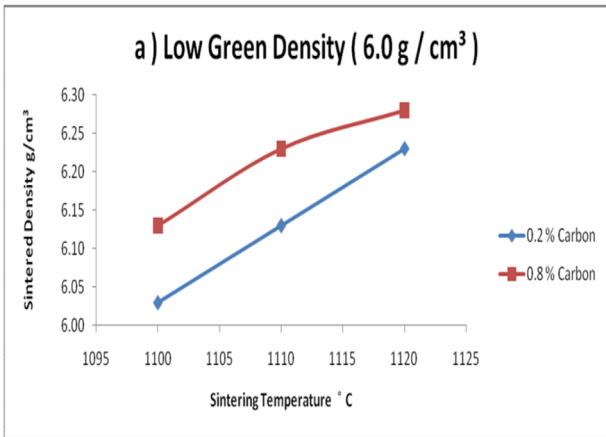
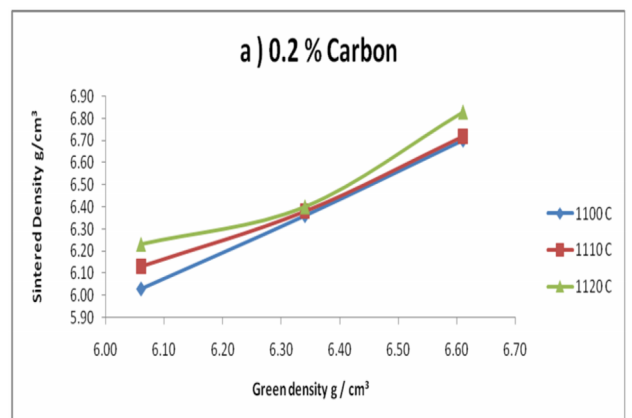


Fig.6 (a, b, and c) : Sintered density versus Sintering temperature for three different green densities

Fig.7. (a, b, and c) : Sintered density versus % Carbon for three different green densities



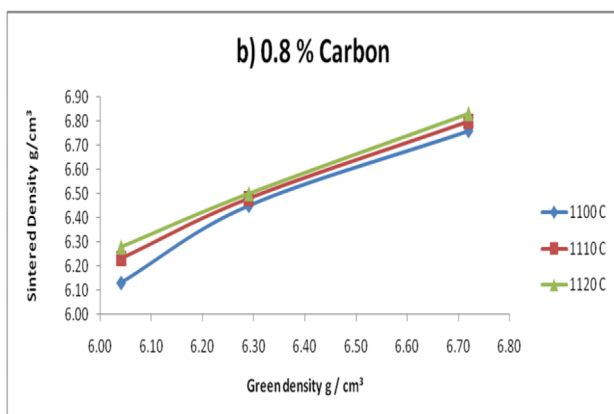


Fig.8. (a, b) : Sintered density versus green density for three different green densities

Table 5. Relative effects of the three process parameters on the dimensional change of Atomised iron powder

Controlling parameter	A change In the Controlling Parameter by	Leads to a change in % Dimensional change Green to sinter by			Estimates Based on
Sintering Temperature	10 ° C	0.0085	0.012	0.002	Figures 3(a, b, c) 0.8 % C
		Low G D	Med G D	High G D	
Carbon Content	0.1 %	0.026	0.028	0.0245	Figures 4 (a, b, c) 1110° C Temp
		Low G D	Med G D	High G D	
Green Density	0.1g /cm ³	0.0049	-	0.0143	Figures 5 (a, b, c) 1110° C temp
		High Carbon	-	Low Carbon	

Multiple linear regression of the data in Table 4 were used to obtain the following relationships between the dependent parameters (Dimensional change – green to sinter and sintered density) and the independent variables (Green density, Carbon Content and Sintering Temperature).

Dimensional Change – Green to sinter (%) = 0.784 + (0.0570 x Green density) – (0.253 x % Carbon) – (0.000708 x Sintering Temperature in ° C)

Sintered Density (g / cm³) = 2.88 + (0.821 x Green density) + (0.135 x % Carbon) – (0.00158 x Sintering Temperature in ° C)

To achieve the desired component dimension, dimensional change – green to sinter can be changed using any of the above three variables, either individually or in any combination. But care should be taken to find the other after-effects of such changes. For example, a decrease in compacting density to achieve the required amount of growth in order to meet the component dimension requirement can proceed to an undesirable decrease in the component's sintered density and hardness.

Similarly, adjustment of the powder properties for achieving a target dimensional change has also some drawbacks. The powder manufacturing process cannot be controlled to produce powder in a very narrow range from lot-to-lot. Typically, the lot-to-lot powder can be about +/- 5% from a target. If frequent adjustments in the % of chemical composition is made during the process, then some of the other properties (such as, apparent density, flow rate, compressibility, and green strength) of the powder will shift to some extent, and this can lead to undesirable effects.

Altering the % of carbon, green density and sintering temperature is the most precise way of modifying dimensional change. For small changes in the sintering temperature (< 10 ° C), the consequences, i.e. changes in the sintered density and mechanical properties, would be negligible but could be estimated. Based on these results it is suggested that altering of dimensional change is best accomplished by regulating the green density, sintering temperature and % of carbon.

It is quiet eminent that meager variations in green density from part-to-part within a given production run can lead to undesirable variations in dimensional change, as noted in Table 5. This study did not consider the effect of change in sintering time, variable cooling rate after sintering, belt speed and fines content of the powder.

The best way to derive good dimensional accuracy is to first choose the powder and the critical process parameter (Apparent density, Flow rate, green density, % of carbon, % of copper, sintering temperature and sintering time). Next step is to make a sintering run with green test specimens made from the chosen powder using the selected process parameters. The dimensional change exhibited in the test run should then be used to design the compaction tooling. Since the powder content of the subsequent production lots may alter by as much as +/- 5 % from the target, small modifications to the sintering temperature should be made for each production run in order to adjust for any shift in dimensional arising from the lot-to-lot variation in the powder content.

VI. CONCLUSION

The following observations have been registered from the investigation:

- The material composition and furnace control as well as the compacted density determine growth – shrinkage or no change in dimension of the metal powder compact.
- Since the lot-to-lot density of the compact can vary by as much as (+/-0.05) g/cm³, adjustment of the sintering temperature is essential, from trial to trial, in order to achieve desirable dimensional control. The amount of adjustment in sintering temperature typically would be within + 5 °C.
- The carbon percentage also imply a stronger influence on dimensional change in atomised iron powder. The growth is greater at lower carbon levels.

VII. ACKNOWLEDGEMENT

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