Sintered Tolerances and the Concomitant Demands on Green Body Homogeneity

Randall M. German, Pavan Suri, Deborah Blaine, and Louis Campbell
Center for Innovative Sintered Products
147 Research West
Pennsylvania State University
University Park, PA 16802-6809 USA

Abstract

Sintered materials are being projected into applications with total dimensional variations of 5 to 15 micrometers, for used in fiber optics, metal cutting, automotive fuel injectors, computer peripherals, transmission gears, and biomedical implants. These dimensional targets greatly tax the current production routes. Accordingly, new insight is needed in how to operate and control production processes to ensure net-shaping with tight dimensional control. This study invokes a mathematical link between green body mass homogeneity and sintered component dimensional scatter. In light of the model, data from die pressed and injection molded powders are analyzed for green mass variations and sintered size variations. The model provides guidelines for assessing research needs for process control, green body inspection, and computer simulation with respect to dimensional tolerance goals. Without attention to an improved infrastructure, the only means to hold tight tolerances is through secondary operations.

Introduction

Computer simulation of sintering has historically focused on calculating the isothermal growth of necks between contacting spherical particles.[1] In recent years these outputs have been used to predict dimensional change, density, sintered strength, surface area loss, thermal conductivity, or other performance factors.[1-14] Because the simulations are inaccurate, computer solutions have not had much impact on dimensional control. Smith[15] has approached this problem from a neural network approach, and for now that is about the best possible method.

Several factors explain the disparity between computer simulations and practice with respect to dimensional control. Most simulations assume monosized, spherical powders of a single composition, ignore the polymer phase and its loss during heating, and even ignore the details or the heating cycles. On the other hand, sintered products rely on -

- wide particle size distributions

- nonspherical and sometimes porous or agglomerated powders
- high polymer contents (either a binder or lubricant)
- nonisothermal heating cycles
- phase transformations during processing
- mixed powders of differing compositions (that melt or dissolve during heating)
- significant atmosphere interactions
- constant process adaptations.

Because of process adaptions, there really is not a constant process that can be modeled to predict final dimensions to a high precision. Further, there are many areas where our basic knowledge is incomplete. For example, density gradients associated with die wall friction affect dimensional tolerances, yet we have little knowledge on how friction changes during compaction, or between powder lots, presses, tool sets, or even during tool frictional heating. Accordingly, improved models will be needed to make accurate predictions of final dimensions. The overarching goal is known as the inverse problem - using the final product definition to derive a specification for the powder, tooling, forming cycle, and heating cycle needed to deliver the specified product.[4,16-18]

Problem Statement

This analysis looks at mass variation as a source of sintered dimension variation. The approach assumes other factors that cause a loss of precision have been eliminated, including:

- elimination of density gradients in the green body
- elimination of thermal gradients from aggressive heating cycles
- elimination of internal vapor stresses during polymer burnout
- elimination of gravity-induced gradients due to substrate friction or improper support.

Accordingly, in the absence of warpage the producer is still faced with two issues:

- prediction of final size to **center** the dimensions
- control of the scatter to hold production within the **tolerance** range.

Industrial components are often specified to a tight clustering around the centered dimension. In many cases the allowed dimensional tolerance zone is ±40 to 150 µm (plus and minus 3 or 6 standard deviations, depending on process yield). This dimensional tolerance zone is tighter in applications associated with sintered cutting tools, fiber optic connectors, microelectronic packages, automotive drive trains, fuel injectors, and hydraulic fluid control; in some cases approaching ±5 µm. In contrast, the ferrous press and sinter industry has a typical dimensional capability (6 standard deviations) of ±135 µm in the pressing direction and ±25 µm perpendicular to the pressing direction.[19] Obviously there is a mismatch between industry capability and emerging user needs.

For this presentation the coefficient of variation will be used to express the normalized variation in mass or size; it is defined as the standard deviation divided by the mean, given as a percent. This parameter is independent of the component and its specification.

Tolerance Sensitivity to Density

Powder shaping processes are good at replicating the tool size. Green dimensions often have low scatter, in the range of a few micrometers, yet sintered components show a much larger dimensional variation. To analyze the problem, the following parameters are used, where the subscript G represents the green condition and the subscript S represents the sintered condition:

- L = mean dimension
- ΔL = dimensional change from green to sintered size
- $\Delta L/L_G$ = shrinkage, sintering dimensional change divided by the green size
- δ = specified tolerance on the sintered dimension L_S
- M = mass
- V = volume
- ρ = fractional density
- σ = standard deviation
- C_V = coefficient of variation (standard deviation divided by mean)

Binder and lubricant masses are ignored in calculating the green density ρ_G, since they burn out in sintering. Assume isotropic shrinkage to simplify the mathematics, without significantly changing the key concepts. The relation between sintering shrinkage $\Delta L/L_G$, green density ρ_G, and sintered density ρ_S is:[20]

$$\rho_S = \frac{\rho_G}{\left[1 - \frac{\Delta L}{L_G}\right]^3} \tag{1a}$$

Rearranging Eq. 1a gives the sintering shrinkage as a function of the green density divided by the sintered density,

$$\frac{\Delta L}{L_G} = 1 - \left(\frac{\rho_G}{\rho_S}\right)^{1/3} \tag{1b}$$

In situations where the sintered density is nearly constant, the sintering shrinkage is simply a function of the green density, $\Delta L/L_G \sim \rho_G^{1/3}$. Figure 1 plots this behavior for molybdenum sintered at 1900°C for 8 hour, showing the linear relation between shrinkage and green density to the one-third power.[21]

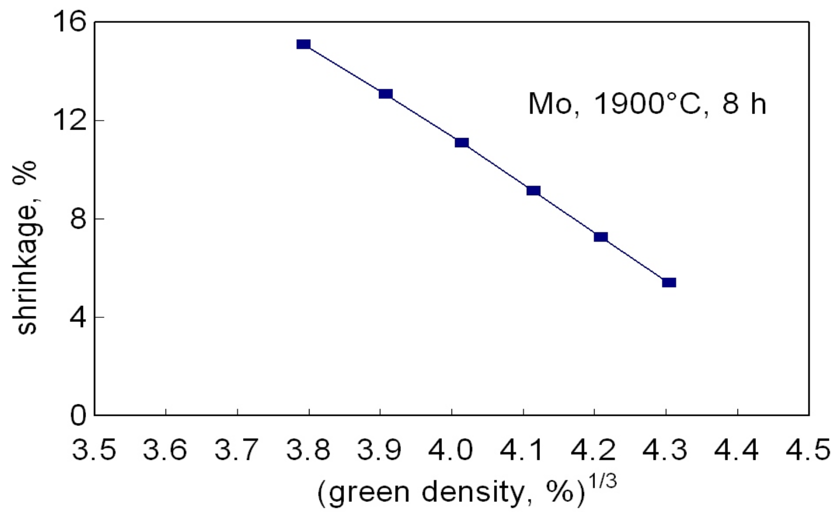

Figure 1. A plot of sintering shrinkage versus fractional green density to the one-third power. The nearly straight line indicates sinter density is not an important factor in final sintered dimensions, at least when the component is well sintered. These data are for a sintered molybdenum powder sintered at 1900°C for 8 h.[21]

Since ΔL is L_G - L_S, it is possible to reorganize Eq. 1b to calculate the sintered size L_S as:

$$L_S = L_G \left(\frac{\rho_G}{\rho_S} \right)^{1/3} \tag{2}$$

Usually the tooling and forming steps give close control on the green size, but the sintered size has more scatter. To assess the factors that might affect the final size, take a partial derivative of Eq. 2 as follows:

$$\partial L_S = \partial L_G \left(\frac{\rho_G}{\rho_S} \right)^{1/3} + \partial \rho_G \frac{L_G}{3\ \rho_S^{1/3} \rho_G^{2/3}} - \partial \rho_S \frac{L_G}{3} \left(\frac{\rho_G}{\rho_S{}^4} \right)^{1/3} \tag{3}$$

This says the sintered dimensional variation ∂L_S has three direct sources - the green size variation ∂L_G, green density variation $\partial \rho_G$, and sintered density variation $\partial \rho_S$. For a well-sintered material $\partial \rho_S$ can be ignored, since grain growth or other microstructure factors limit sintering. If forming is in a single cavity tool, then the green size change ∂L_G should be small. Consequently, we focus our attention on the green density scatter. Density is mass over volume, and since the green volume is well controlled,

$$\rho_G = \frac{M_G}{\rho_O \; V_G} \qquad so \qquad \partial\rho_G = \frac{\partial M_G}{\rho_O \; V_G} \tag{4}$$

where M_G is the green mass, V_G is the green volume (assumed constant), and ρ_O is the theoretical density of the material. Combining Eqs. 3 and 4 gives a relation between green mass variation and sintered dimension variation.

Statistical Analysis

An audit of several powder injection molding (PIM) studies[22] found the typical coefficient of variation in sintered dimensions was 0.22%. For example, Cardamone[23] examined injection molded tungsten heavy alloys for dimensional scatter using seven dimensions while including factors such as day-to-day variations. Her results showed dimensional variations in the green bodies had a typical coefficient of variation of 0.04%, but the green mass had a 0.1% coefficient of variation. After sintering the size coefficient of variation increased fivefold, averaging 0.2%, suggesting the green mass variation amplified the scatter in sintered size.

Along these lines, Piemme[24] performed experiments using powder injection molded 316L stainless steel formed into a hexagonal screwdriver blade holder. The measurements included green, debound, and sintered dimensions on several features, with variations in feedstock and nine different molding conditions (typically 75 measured parts for each combination). The mean green mass was 17.55 g which included 1.068 g of binder (6.08%). For a green length of 44.7 mm the size change in sintering to 7.75 g/cm^3 was 14.3%, giving a final length of 38.2 mm. The standard deviation in sintered size was 37 µm, corresponding to a 0.1% coefficient of variation. His data were analyzed for correlations giving the results summarized in Table 1, showing a significant relation between sintered size variation and green mass variation. Table 2 compares the size variations green and sintered for the molding conditions giving the lowest and highest green mass variations. The green size variations are the same, yet the sintered size variation is three time larger for the higher mass variation condition. Regression analysis shows that 77% of the sintered size variation is explained by the green mass and green length variations. The remaining sintered size variation comes from the sintering run, furnace position, measurement error, debinding, and such. Thus, sintered size variation is dominated by variations in green length and green mass. In this study, green length variations were induced by systematic changes in molding conditions; normally only mass variations would occur in production.

Table 1. Correlations for Statistically Significant Relations (all greater than 95% significant)

sintered mass and green mass - 0.957
sintered size and green mass - 0.855
sintered size and sintered mass - 0.800
sintered size variation and green mass variation - 0.874
sintered size variation and sintered mass variation - 0.783

Table 2. Comparison of High and Low Green Mass Variation Conditions

molding condition	**C_V mass, %**	**C_V molded size, %**	**C_V sintered size, %**
lowest green mass scatter	0.052	0.016	0.099
highest green mass scatter	0.223	0.016	0.300

Simplified Model

The above data suggest green mass variation is a significant factor with respect to controlling sintered dimensions. Accordingly, a simplified model is possible. The sintered size variation ∂L_S dependence on green density variation $\partial \rho_G$ is simplified by realizing the green volume is usually controlled by the tooling, yet the green mass is variable, giving

$$\partial L_S = \partial \rho_G \frac{L_G}{3\,(\rho_G^2\,\rho_S)^{1/3}} = \partial M_G \frac{L_G}{3\,(\rho_O V_G)^{1/3}\,M_G^{2/3}\,\rho_S^{1/3}} \tag{5}$$

The parameter ∂L_S links the scatter in sintered size to variations in green mass ∂M_G. The theoretical density is constant. To a good approximation the green mass and sintered mass are the same, thus,

$$\partial L_S = \frac{L_G}{3}\left(\frac{V_S}{V_G}\right)^{1/3} \frac{\partial M_G}{M_G} \tag{6}$$

Since we assume the green and sinter mass are the same, then the density ratio in Eq. 2 is effectively the inverse volume ratio and Eq. 6 simplifies to give,

$$\frac{\partial L_S}{L_S} = \frac{1}{3}\,\frac{\partial M_G}{M_G} \tag{7}$$

Equation 7 says the normalized sintered size variation is proportional to the normalized green mass variation. Accordingly, goals for tight sintered tolerances can be assessed based on green mass control capabilities. In other words, the green mass variation and sintered size variation follow a simple relation,

$$\frac{green\ mass\ variation}{mean\ mass} \leq 3\ \frac{tolerance}{mean\ size} \tag{8}$$

For any mean size and tolerance the maximum allowed green mass variation in production is automatically set. The actual allowed green mass variation must be less than the value calculated using Eq. 8 to allow for other factors that contribute to dimensional variation. So this is an upper bound constraint on production, yet is helpful in assessing options.

Implications and Applications

As an example of using Eq. 3, assume the situation involves die compaction in a mechanical press to a fixed final volume, such that the green size is tightly controlled. Ignoring the polymer mass, let the fractional green density be 0.92 and the sintered fractional density be 0.93, corresponding to a sintering dimensional change of 0.36%. If the tolerance is specified at ±0.2%, then the allowed total variation in green mass would be ±0.6%, assuming no other sources of dimensional variation.

Consider this variation in light of powder metallurgy (P/M) industry data[25-27] that indicate dimensional tolerances typically range from ±20 to 150 µm. Semel[28] showed data reflecting a 0.42% C_V in mass using standard iron powder and 0.13% C_V for binder treated powder. Schneider *et al.*[29] published data on powder-forged connecting rods, showing the dimensional variation in nine dimensions. Dimensions are held to tolerance ranges from ±0.14% to ± 0.20% while mass variation was typically less than 0.2%. According to Eq. 8, the part mass variation is compatible with the dimensional tolerance range. Thus, various reports on tolerances and mass variation show compatibility with Eq. 8.

Upadhyaya *et al.*[30] measured mass and sintered size for spray dried cemented carbide powders pressed into cutting inserts and vacuum sintered. The green mass C_V was 0.16% with a corresponding ±23 µm sintered dimension variation. From the same compaction run, samples were selected to reduce the mass variation. The sorted samples had a 0.12% mass variation which resulted in a smaller ±15 µm sintered dimension variation; A reduction in mass variation produced a corresponding reduction in dimensional variation.

In PIM there are several sources of green mass variation. One is the feedstock and its homogeneity. Data on molded part mass variations show C_V in the 0.1% to 0.3% range.[23,24,31-33] The lower values are associated with closed-loop pressure cavity control. A 0.1% to 0.3% mass C_V suggests a ±3 standard deviations dimensional precision in the same 0.1 to 0.3% range with no other sources of dimensional variation. Many PIM firms advertise precision at ±0.3% to 0.5%,[22] which is compatible with this mass variation. One problem is with the use of multiple-cavity tooling, where systematic cavity-to-cavity variations occur in terms of filling, cooling, or mold dimensions. Such variations consume the sintered tolerance budget and require even tighter mass uniformity.

Discussion

The relation provided by Eq. 8 seems reasonable based on a few studies reporting data on green mass variations and sintered size variations. The model provides a first basis for analyzing if product goals are compatible with process capabilities. An alternative expression to the coefficient of variation is the process capability C_P, defined as the tolerance range ±δ divided by three times the standard deviation. Unless the process mean tolerance is centered in the tolerance range, the process control capability C_{Pk} is smaller. The current model is equally applicable to these measures of dimensional scatter using the transform $C_P = \delta/(3\ L_S\ C_V)$.

Mass is a low-cost, nondestructive monitor for green body variations. As P/M encounters tight dimensional tolerances the response is to use post-sintering deformation or machining. An alternative is through reduced green mass variations via more homogeneous powders, powder delivery systems, presses, and tooling. A related issue is on-line inspection. For example nondestructive testing techniques, such as ultrasonic velocity measurements, can only detect 1% density gradients.[34] Based on Eq. 8, these are not sensitive enough to significantly improve dimensional precision beyond current capabilities.

Computer simulations of P/M processes are inaccurate in predicted size because they do not have good models nor sufficiently accurate verification data. For example, in die pressing the powder-tool friction varies during the compaction stroke and even varies by a factor of 2 between presses.[35] Unfortunately the simulations assume constant friction. Consequently, the simulated green gradients are not accurate, so the sintered size predictions are not to the required accuracy. Complicating the problem is the general trend toward tighter tolerances for sintered components. Tighter tolerances require more uniform green bodies. At this point, a fruitful route to improved sintered dimensional precision is by focused efforts to reduce green mass variations.

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