Green Body Homogeneity Effects on Sintered Tolerances

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Abstract

The link between green body heterogeneity and sintered tolerances is expressed in a generic form. Data from die compaction and injection molding of stainless steel, steel, tungsten carbide, and other materials are examined to find the dominant green mass variation effect. Calculations are used to assess goals for process control, inspection, and computer simulations in light of contemporary dimensional tolerances. The findings show that current dimensional control goals exceed current capabilities. Hence secondary operations will continue to be the best means for holding dimensional scatter in target ranges.

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 and other performance factors.[1-14] Because the simulations are inaccurate, computer solutions have not had much impact on dimensional control. Consequently, Smith[15] has approached this problem from a neural network approach. Improved models are 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, process, and tooling.[4,16-18]

Problem Statement

This analysis looks at mass variation as a source of sintered dimension variation. 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 :m (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 :m. In contrast, the ferrous press and sinter industry has a typical dimensional capability (6 standard deviations) of ±135 :m in the pressing direction and ±25 :m perpendicular to the pressing direction.[19] A mismatch occurs with respect to 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.

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, *)L* = dimensional change from green to sintered size, *)L/*L_G = shrinkage, sintering dimensional change divided by the green size, * = specified tolerance on the sintered dimension L_S, M = mass, V = volume, ρ = fractional density, σ = standard deviation, and 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 *)L/*L_G, green density ρ_G, and sintered density ρ_S is:[20]

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

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

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

In situations where the sintered density is nearly constant, then *)L/*$L_G \sim \rho_G^{1/3}$.[21]

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

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

Usually the tooling and forming steps give close control on the green size, but the sintered size has more scatter. To determine controlling factors, take a partial derivative of Eq. 2:

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

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 is small. Consequently, we focus on the green density scatter. Density is mass over volume, and green volume is controlled,

$$\rho_G = \frac{M_G}{\rho_O V_G} \quad \text{so} \quad \partial\rho_G = \frac{\partial M_G}{\rho_O V_G} \qquad (4)$$

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. Eqs. 3 and 4 relate 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 scatter increased 5x, 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 formed into a hexagonal screwdriver blade holder. The measurements included green, debound, and sintered dimensions on several features, with variations in feedstock and 9 different molding conditions. 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 :m, corresponding to a 0.1% coefficient of variation. His data gave 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 3x 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.

Table 1. Correlations for Statistically Significant Relations (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 $\partial \Delta_G$ is simplified by realizing the green volume is usually controlled by the tooling, yet the green mass is variable, giving

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

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

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

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,

$$\frac{\partial L_S}{L_S} = \frac{1}{3}\frac{\partial M_G}{M_G} \qquad (7)$$

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. The green mass variation and sintered size variation show,

green mass variation/green mass # 3 tolerance/size (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

Consider industrial size variation data[25-27] that indicate dimensional tolerances typically range from ±20 to 150 :m. 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, the various reports are compatible with Eq. 8.

Upadhyaya *et al.*.[30] measured mass and sintered size cemented carbide pressed into cutting inserts and vacuum sintered. The green mass C_V was 0.16% with a corresponding ±23 :m 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 :m sintered dimension variation; a reduction in mass variation produced a corresponding reduction in dimensional variation.

In PIM, 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 have precision of ±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 tolerance budget and require mass uniformity.

Discussion

Eq. 8 is 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. 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; ultrasonic velocity measurements can only detect 1% density gradients.[34] Based on Eq. 8, this will not be sufficient to improve dimensional precision beyond current capabilities.

Computer simulations of P/M processes are inaccurate in predicted size because they do not have good models or 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.

Acknowledgment

This effort was performed in the Center for Innovative Sintered Products at Penn State under funding from the National Science Foundation via Grant No 0200554, Protocol Development for Net Shape Powder Metal Part Production via Cold Compaction.

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