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# Relationship between Binder Contents and Mechanical Properties of 17-4 Ph Stainless Steel Fabricated By PIM Process and Sintering

**K. A. Khalil and Sug Won Kim***

Division of Advanced Materials Engineering, RIAMD, Chonbuk National University,
664-14, Deokjindong 1-ga, Deokjin-gu, Jeonju-si, Jeonbuk 561-756, Korea

Mechanical properties and microstructures of 17-4 ph stainless steel parts produced using different binder contents (powder loading) of powder injection molding (PIM) feedstock have been studied. The tensile and wear properties have been evaluated. Wear tests were conducted by a pin-on-disk tribometer, without lubricant, at different loads and sliding distance. SEM examination of the fracture surfaces revealed good particle bonding and a high ductile fracture surface for high powder loading. The surface fractures of the bars with higher powder loading show a closed porosity. High performance properties such as fully dense, ultimate tensile strength, hardness and wear resistance are obtained with high powder loading.

**Keywords:** dental brackets, wear resistance, fracture properties, powder-binder characteristics, 17-4 ph stainless steel

## 1. INTRODUCTION

17-4 ph stainless steel is used for many devices requiring a combination of good corrosion, wear resistance, and high strength such as dental brackets, gun parts, surgical instruments, and aerospace industries. Thus far, the growth technology of Powder Injection Molding (PIM) has been employed for manufacturing complex shaped, high volume metal or alloy parts [1]. PIM can be described as follows: powder of metals or ceramics are mixed with a binder to make a feedstock that is injection-molded into a die of required shape. Careful removal of the plastic binder (using solvent decomposition and/or thermal decomposition) leaves a skeleton of metal that can be sintered traditionally. The PIM process offers several advantages over the conventional powder compaction process. The products prepared by the PIM process can avoid the density gradient obtained in the conventional press/sinter process [2]. Gas or water atomized stainless steel powders, which are shaped and processed via injection molding, can achieve high complexity of part geometry with mechanical and corrosion properties that are similar or superior to those of wrought material [3].

There have been numerous reports on the optimal molding conditions for attaining good moldability and avoiding distortion during debinding [4-28]. However, there is less published data in the literature on the effects of PIM powder-binder mixture characteristics on the fracture properties and wear resistance. Fox [9] studied warpage and fracture in green parts arising from residual stresses created during cooling. It was concluded that the green part exhibits viscoelastic behavior during cooling and that cooling-generated stresses lead to internal fractures and asymmetric cooling leads to warpage. The mechanical behavior of compacts at temperatures typical for molding and the initial steps of thermal debinding were studied by Moller and Lee [10]. It was found that the feedstock is not purely viscous at the molding and initial thermal debinding temperature, but rather the feedstock displays a combination of viscous, elastic, and plastic behavior. Kostic *et al.* [11] found that the stress distributions are strongly influenced by the control sprue solidification time. In addition, steep changes in pressure in molding during the course of solidification, which widen the residual stress distribution, were observed.

17-4 ph stainless steels are used in applications demanding general corrosion resistance at room or moderate operating temperatures. However, their use is often limited by their relative softness and susceptibility to wear [23]. The aim of the present work is to study the effects of PIM powder-binder mixture characteristics on the fracture properties and wear resistance of 17-4 ph stainless steel compacts. The present investigation deals with the dry sliding wear behavior of this material when used in dental brackets and made by a PIM process.

## 2. EXPEREMENTAL PROCEDURE

A gas atomized 17-4 ph stainless steel powder, supplied by

*Corresponding author: ksw@chonbuk.ac.kr

BASF Germany, with a particle size distribution of $d_{10}$ = 5 μm, $d_{50}$ = 12 μm, $d_{80}$ = 22 μm, and 12 μm average particle size have been used in the present investigation. Apparent and tap densities of the powder are 3.92 g/cm$^3$ and 4.70 g/cm$^3$, respectively. Paraffin wax was used as the major binder component. The minor components were Ethylene Vinyl Acetate (EVA) and High-Density Polyethylene (HDPE). Stearic Acid (SA) was included as a surface-active agent.

The powders were hot mixed together with thermoplastic binders using a LH60 Roller mixer for 2 h so as to produce a homogeneous feedstock. This mixture was then cooled and finely granulated using LSJ20 plastic extruder. Four powder-binder mixtures with different powder contents were prepared to investigate the effect of powder loading (the concentration of the powder in the binder media). The feedstock was injected into an SZ-28/250 injection-molding machine. A schematic diagram of the PIM process is presented in Fig. 1.

Application of a solvent followed by hydrogen atmosphere thermal debinding was used to remove the binder system. The molded compacts containing both the polymer and the powder were initially weighed. The samples were debound in heptane so as to remove the wax, the only soluble component of the binder system, thus leaving pore channels. These channels provide pathways for the decomposed constituent to escape to the ambient during subsequent thermal debinding. Samples were removed from the solvent at regular intervals and dried at 40 °C in order to remove the absorbed moisture. The dried samples were weighed, and the weight loss of the binder system was subsequently determined. Thermal debinding was performed in a hydrogen atmosphere. Sintering was carried out in a vacuum atmosphere at 1380 °C for 1.5 h.

PIM tensile specimens (40 mm long and 5 mm diameter) were used in this study. Tensile testing was carried out under a constant crosshead speed of 0.635 mm/min. After tensile testing the fracture surface was prepared for fractography. Wear resistance was evaluated using a pin-on-disk wear testing machine. The disk is made of AISI 5190 steel with 62 HRc hardness whereas the pin is comprised stainless 17-4 ph steel sintered parts. Wear tests were performed under different loads and sliding distances, at a sliding velocity of 0.2 m/s. Tests were conducted at room temperature without lubricant. The wear resistance was measured in terms of weight loss of the tested materials using a precision balance with a resolution of 0.1 mg. A Vickers indenter with a load of 10 kg was employed to determine the hardness of the sintered samples. The size of the indentation cracks was used to determine the fracture toughness ($K_{IC}$) of the sample. Each Vickers impression presented two pairs of radial cracks emerging from the corners. A total of ten pairs of cracks were obtained. For calculations, some pairs of cracks were used for each sample. The equation used to determine the $K_{IC}$ values is as follows: Eq. 1 [29].

$$K_{IC} = 0.016\left(\frac{E}{H}\right)^{1/2}\frac{P}{C^{3/2}} \quad (1)$$

where $K_{IC}$ = fracture toughness (MPa. M$^{1/2}$), P = applied load (N), E = Young's modulus (GPa), H = hardness (GPa), and C = diagonal crack length (mm).

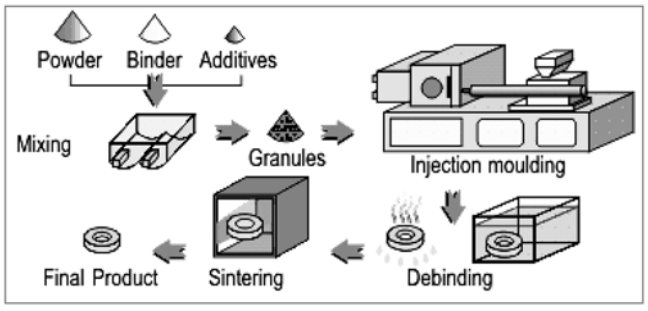


**Fig. 1.** Schematic diagram of PIM process.

## 3. RESULTS AND DISCUSSION

### 3.1. Effect of powder loading on the mechanical properties

The effects of binder content or powder loading on the rheological properties have been studied in detail [2,5,7-16] but there is no data published in the literature on the effect of powder loading on the mechanical properties of the final products. The effect of powder loading on the tensile properties has been studied here. Fig. 2 shows the typical stress - strain curves for compacts molded with different powder content (powder loading). The average result of three tensile tests carried out under the same conditions for each specimen was obtained. The dependence of ultimate tensile strength (average value for three tensile test specimens) on the powder loading is given in Fig. 3. It is clear that the tensile strength increases with increasing powder loading. As the molded part is extremely porous, very large shrinkage occurs in the compacts produced with low powder loading. Increas-

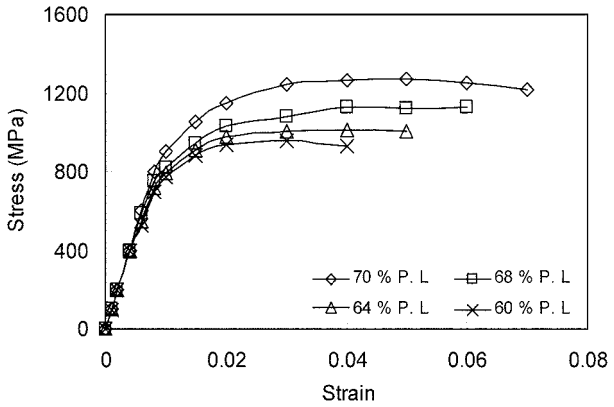


**Fig. 2.** Stress-strain curves for different powder loadings (% P. L).

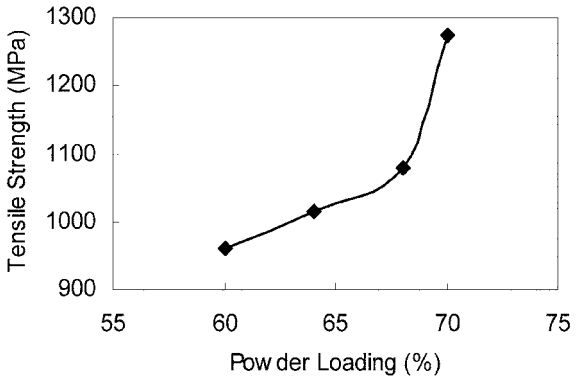


**Fig. 3.** Variation of tensile strength with powder loading.

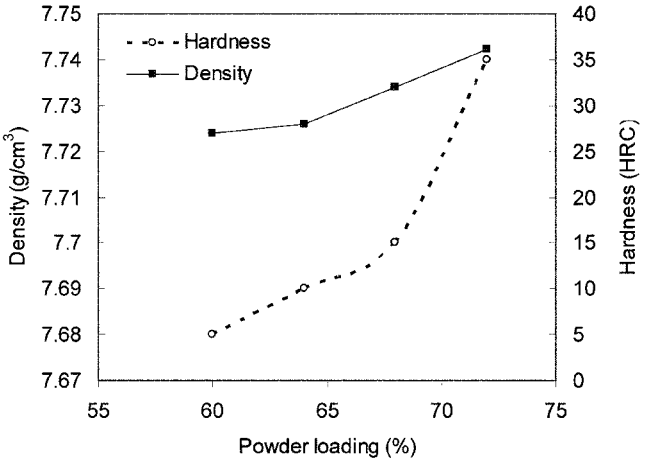


**Fig. 4.** Variation of density and hardness with powder loading.

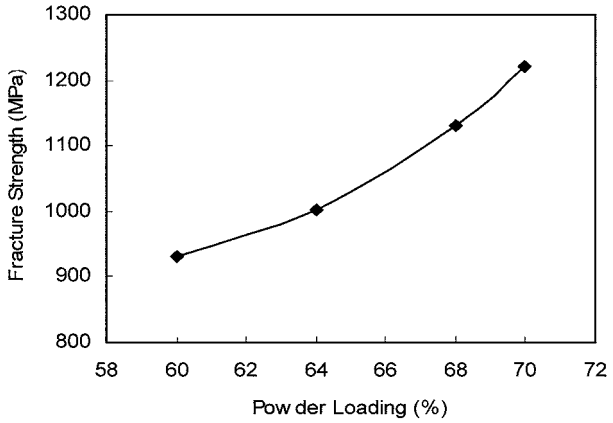


**Fig. 5.** Variation of fracture strength with powder loading.

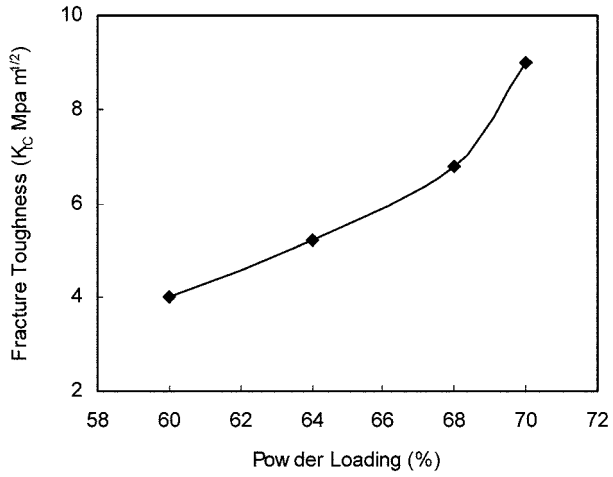


**Fig. 6.** Variation of fracture toughness with powder loading.

ing the powder loading decreases the porosity and increases the strength of the sintered compacts. Also, the powders used in PIM are much finer than those used in PM, and consequently sintering takes place more readily on an account of the higher surface energy of the particles.

Fig. 4 shows the variation in density and hardness of the sintered compacts as a function of the powder loading. As shown in this figure, the density increased with increasing powder loading. This is due to the increase in the molded part porosity with decreasing powder loading. Furthermore, very large shrinkage is required in the compacts with low powder loading, thus causing low density. Increasing the powder loading results in a decrease in the porosity and an increase in the density of the sintered compacts. The hardness of the compact depends on the compact density, and thus higher the compact density yields higher compact hardness. This is clearly indicated in Fig. 4.

Figs. 5 and 6 show the variation of fracture strength and toughness of the sintered compacts as a function of powder loading. As shown, the fracture strength and toughness increase with increasing powder loading due to the increase in the density of the sintered compacts. This is due to the decrease in the molded part porosity with increasing powder loading. In addition, very large shrinkage is required in the compacts with low powder loading, thus causing low density. The hardness of the compact depends on the compact density, and therefore higher compact density yields higher compact hardness and strength. This is clearly indicated in Figs. 3, 4, and 5. Unlike conventional powder metallurgy, powder injection molding employs smaller powders with particle size ranging from 3 to 20 mm. The relative density of the green compacts before sintering is about 60 % of the theoretical value according to the powder loading, and becomes 98 % after sintering. Consequently, the sintering shrinkage is generally above 12 %. The pore size and microstructure are very important factors with respect to the mechanical properties.

### 3.2. Effect of powder loading on fracture surfaces

The fracture surfaces of the sintered 17-4 ph stainless steel compacts with different powder loading and 1380 °C sintering temperature for 90 min are shown in Figs. 7 to 10. These

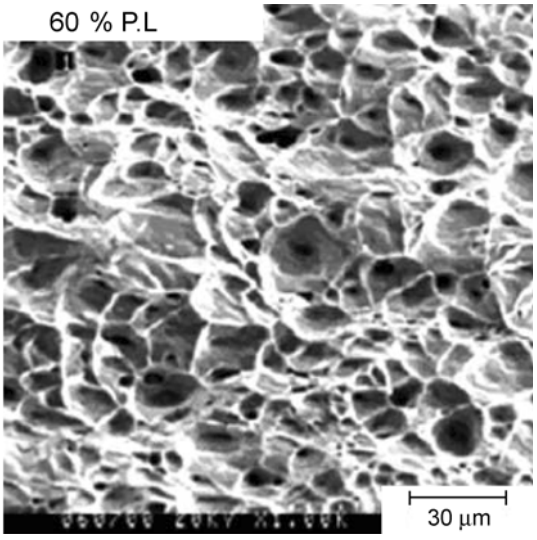


**Fig. 7.** Fracture surface of the sintered compact with 60 % powder loading.

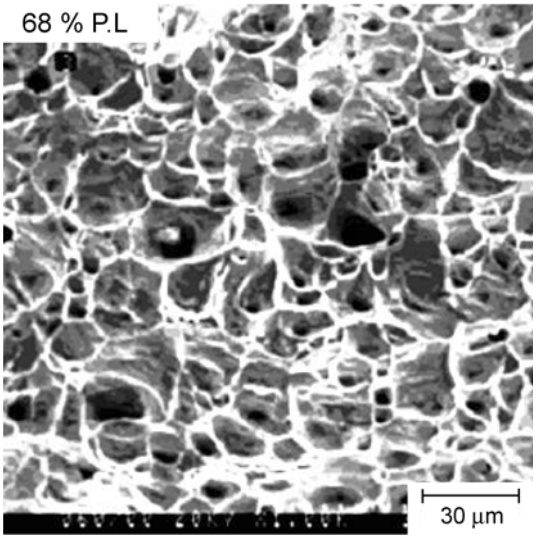


**Fig. 9.** Fracture surface of the sintered compact with 68 % powder loading.

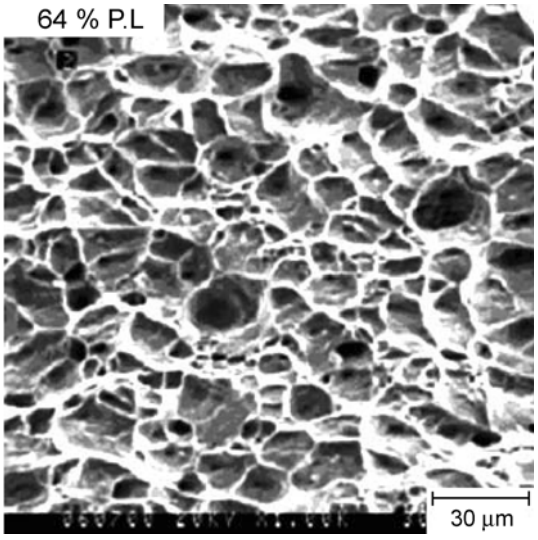


**Fig. 8.** Fracture surface of the sintered compact with 64 % powder loading.

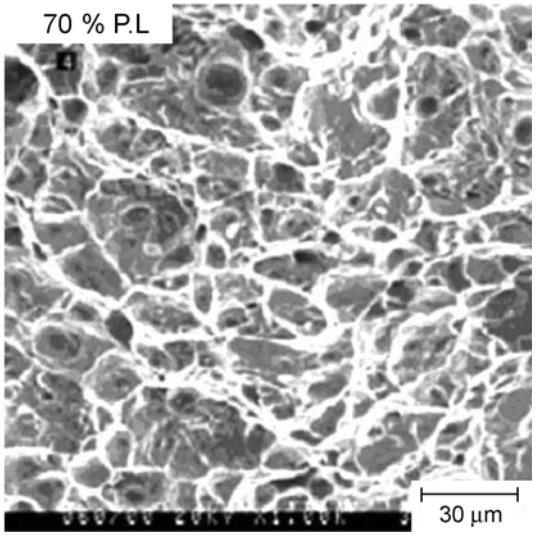


**Fig. 10.** Fracture surface of the sintered compact with 70 % powder loading.

figures show the SEM observations of the fracture surfaces of the tensile test specimens with different powder loading. All specimens were sintered under the same conditions. From these figures it can be concludes that fracture in most of the samples was ductile fracture at the surface. However, the fractures in specimens 7 and 8 occurred along the grain boundary due to lower powder loading. Figs. 9 and 10 exhibit nearly closed porosity due to higher powder loading. In addition, a higher ductile fracture surface can be observed and good particle bonding is noted for high powder loading. Specimens that were molded with low powder loading, meanwhile, exhibit partial particle bonding. Some particles in Figs. 9 and 10 show small bonding and some show only some small sinter nicks. An open porosity is also visible. Increasing the powder loading is thus recommended in order to enhance the mechanical properties of the PIM sintered compacts.

### 3.3. Effect of powder loading on wear resistance

Results of wear tests conducted on four different powder loadings are shown in terms of weight loss-sliding distance and weight loss-load plots in Figs. 11 and 12. The total weight loss-sliding distance and weight loss-applied load curves were constructed by taking the average result of three tests carried out under the same conditions for each specimen. The weight loss of the samples increased linearly with

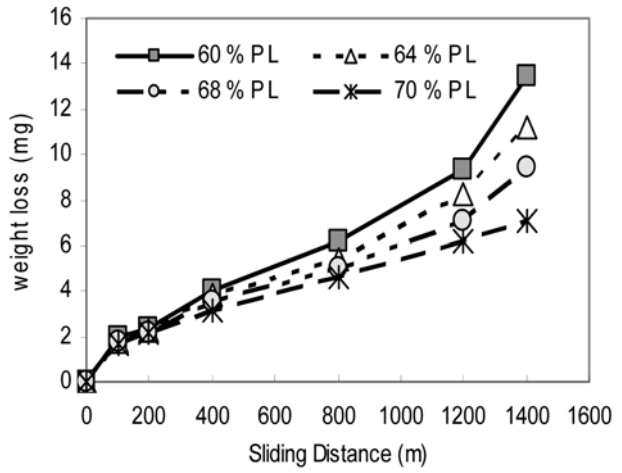


**Fig. 11.** Wear weight loss of 17-4 ph stainless steel for different sliding distances.

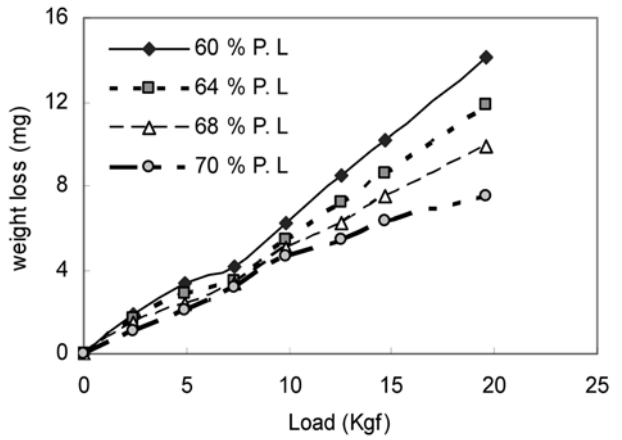


**Fig. 12.** Wear weight loss of 17-4 ph stainless steel for different loading.

increasing sliding distance and loads for the entire range of powder loading investigated here. In most cases, no steady state stage was attained during the wear test period. The results show that the specimens with high powder loading (70 %) are the most wear-resistant. The lowest wear resistance among these specimens was obtained for specimens with low powder loading.

## 4. CONCLUSION

From the present investigation the following conclusion can be obtained: The densification process was almost completed after one and half hours at 1375 °C, reaching a density of 7.71 g/cm$^3$, 98.8 % of the theoretical density. Increasing the powder loading decreases the porosity and increases the tensile strength of the sintered compacts. Good particle bonding, closed porosity, and a higher ductile fracture surface were observed with high powder loading. The wear resistances of the 17-4 ph stainless steal compacts improved with high powder loading. Increasing the powder loading is the optimal means of enhancing the mechanical properties and wear resistance of the PIM sintered compacts.

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## REFERENCES

1. M. German, *Powder Injection Molding*, Metal Powder Industries Federation, Princeton, NJ (1990).
2. Y. M. Li, Q. Xuanhui, H. Baiyun, and Q. Guanghan, *Trans. Nonferrous Met. Soc. China* **7**, 103 (1997).
3. R. P. Koseskia, P. Suria, *et al.*, *Mater. Sci. Eng. A* **390**, 171 (2005).
4. Y. M. Li, X. H. Qu, L. Zhilin, and H. Baiyun, *Trans. Nonferrous Met. Soc. China* **8**, 576 (1998).
5. Y. M. Li, Q. X. Hui, and H. Baiyun, *Ph.D. Thesis*, Powder Metallurgy Institute, Central South University, Changsha, Hunan, P. R. China (1998).
6. M. Jinying and Q. Xuanhui, *M. Sc. Thesis*, Powder Metallurgy Institute, Central South University, Changsha, Hunan, P. R. China (1999).
7. Y. M. Li, H. Baiyun, and X. Qu, *Int. J. Of Science and Practice of Powder Metallurgy* **42**, 86 (1999).
8. B. O. Rhee and C. I. Chung, *Powder Injection Molding Symposium* (eds., P. H. Booker, J. Gaspervich, and R. M. German), p. 131, MPIF, Princeton, NJ (1992).
9. R. T. Fox and L. Daeyoung, *Int. J. of Powder Metall.* **30**, 221 (1994).
10. J. C. Moller and D. Lee, *Int. J. Powder Metall.* **30**, 103 (1994).
11. B. Kostic, T. Zhang, and J. R. G. Evans, *Int. J. Powder Metall.* **29**, 251 (1993).
12. L. Bogan, N. Amoroso, and R. Einhorn, *Advances in Powder Metallurgy and Particulate Materials* (eds., T. M. Cadle and K. S. Narasimhan), Vol. 19, p. 265, Metal Powder Industries Federation, Princeton, NJ (1996).
13. R. Raman, W. Slike, and R. M. German, *Advances in Powder Metallurgy and Particulate Materials* (eds., A. Lawley and A. Swanson), Vol. 5, p. 1, Metal Powder Industries Federation, Princeton, NJ (1993).
14. J. A. Horn and B. R. Patterson, *Advances in Powder Metallurgy and Particulate Materials* (eds., A. Lawley and A. Swanson), Vol. 5, p. 17, Metal Powder Industries Federation, Princeton, NJ (1993).
15. C. M. Wang, R. L. Leonard, and T. J. McCabe, *Advances in Powder Metallurgy and Particulate Materials* (eds., A. Lawley and A. Swanson), Vol. 5, p. 31, Metal Powder Industries Federation, Princeton, NJ (1993).
16. M. J. Rosner, X. Zhang, M. Kojima, R. A. Posteraro, and J.

T. Lindt, *Powder Injection Molding Symposium* (eds., P. H. Booker Gaspervich and R. M. German), p. 451, MPIF, Princeton, NJ (1992).
17. Z. Haorong, R. M. German, and A. Bose, *J. of Powder Metall.* **26**, 217 (1990).
18. K. S. Hwang and T. H. Tsou, *Metallurgical Transaction A* **23**, 2775 (1992).
19. K. N. Hunt, J. R. G. Evans, and J. Woodthorpe, *J. of Materials Science* **26**, 292 (1991).
20. K. N Hunt, J. R. G. Evans, and J. Woodthorpe, *J. of Materials Science* **26**, 2229 (1991).
21. L. Shaojun, Huan Baiyun *et al.*, *Trans. of Nonferrous Met. Soc. China* **9**, 338 (1999).
22. F. Jinglian, H. Baiyun, *et al.*, *Trans. of Nonferrous Met. Soc. China* **9**, 93 (1999).
23. M. Vardavoulias, M. Jeandin, *et al.*, *Tribology International* **29**, 499 (1996).
24. R. Supati, N. H. Loh, K. A. Khor, and S. B. Tor, *Materials Letters* **46**, 109 (2000).
25. K. S. Hwang, *Proceeding of Powder Injection Molding Conference* (eds., R. M. German, W. Helmut, and G. C. Robert), p. 173, Innovative Material Solution/Inc., State college, PA (1998).
26. G. R. White and R. M. German, *Advances in Powder Metallurgy and Particulate Materials* (eds., A. Lawley and A. Swanson), p. 101, Metal Powder Industries Federation, Princeton, NJ (1993).
27. R. Steger, *PM'94, Powder Metallurgy World Congress*, Vol. 2, p. 1197, Paris (1994).
28. R. Miura and S. Takamori, *Powder Injection Molding Symposium* (eds., P. H. Booker *et al.*), p. 359, MPIF, Princeton, NJ (1992).
29. G. R. Anstis, P. Chantikul, B. R. Lawn, and D. B. Marshall, *J. of American Ceramic Society* **64**, 533 (1981).