

# Factors Controlling the Sintering of Ceramic Particulate Composites: II, Coated Inclusion Particles

Ching-Li Hu and Mohamed N. Rahaman\*

Ceramic Engineering Department, University of Missouri, Rolla, Missouri 65401

Composite powders were synthesized by coating coarse  $ZrO_2$  inclusion particles with a cladding of fine-grained, crystalline  $ZnO$  powder using a chemical precipitation technique. Three different inclusion sizes (1, 3, and 14  $\mu m$ ) were used by selecting the size of the starting  $ZrO_2$  powder, and the volume fraction of the inclusions was controlled by the amount of  $ZnO$  precipitated. The powders were compacted by uniaxial pressing in a die and then sintered at a constant heating rate of  $4^\circ C/min$  to  $1500^\circ C$ . The sintering kinetics were almost independent of the inclusion volume fraction, and of the inclusion size, for inclusion contents up to  $\approx 40$  vol%. Furthermore, composites containing up to  $\approx 40$  vol% inclusions were sintered to almost full density under the same conditions used for the unreinforced matrix. This is considerably better than the densities obtained for conventionally mixed powders, where a modest inclusion content ( $< \approx 10$  vol%) has been observed to cause a severe reduction in the sintered density of the composite matrix. The kinetic data and microstructural observations are a further indication that the main factors which oppose the free sintering of ceramic particulate composites are processing-related; these factors are (i) inclusion-inclusion interactions which constrain the matrix and (ii) the packing of the matrix phase in regions immediately surrounding the inclusions.

## I. Introduction

THE results of the preceding paper<sup>1</sup> indicated that the main factors controlling the conventional, pressureless sintering of polycrystalline ceramic-matrix composites were (i) a constraint on the matrix produced by the interacting inclusion particles and (ii) the packing of the matrix phase, especially in regions immediately surrounding the inclusion particles. The composites were formed by two different methods: (i) by mixing the matrix and inclusion powders conventionally in a ball-mill, followed by pressing the dried powder mixture uniaxially in a die, and (ii) by dispersing the matrix and inclusion powders to produce a stabilized slurry followed by slip casting. The matrix phase was fine-grained  $ZnO$  powder and the inclusions were coarse, inert  $ZrO_2$ . Both of these forming methods would be expected to produce a randomly distributed inclusion phase within the matrix. Although the slip-casting method produced a more uniform matrix packing, nevertheless it did not lead to a significant improvement in the composite sinterability.

The aim of the work described in the present paper was to investigate whether the sinterability of polycrystalline matrix composites could be improved if the problems associated with the matrix packing and the interacting inclusions were alleviated. In the present investigation, composite powders

were formed by a novel technique which involved coating the individual inclusion particles ( $ZrO_2$ ) with a cladding of the matrix powder ( $ZnO$ ) by chemical precipitation from a solution of a zinc salt. The synthesized coated powders were collected and consolidated by pressing in a die to form the green bodies.

The coating process used for synthesizing the composite powders is expected to lead to a number of beneficial sintering characteristics, compared to composites formed by more conventional techniques (e.g., consolidation of mixed powders). Figure 1 shows schematic diagrams of the compacted bodies formed from (a) coated inclusion particles and (b) conventional mixing of the inclusions and the matrix powder. For uniformly coated inclusions, the compacted body would be expected to have a uniform distribution of the inclusion particles, compared to the body formed from conventionally mixed powders. The packing of the matrix powder would also be expected to be more uniform for the coated inclusion particles, especially in the regions immediately surrounding the inclusions. Furthermore, gross packing inhomogeneities would be expected to result from clustering of the randomly distributed inclusion particles in the conventionally processed bodies. Finally, direct inclusion-inclusion interactions would be absent or minimized in bodies formed from the coated inclusion particles.

The composites formed from the coated inclusion particles will also provide a better model experimental system for comparison with the predictions of the concentric sphere model,<sup>2-4</sup> which has been used to analyze the sintering of a matrix containing rigid inclusions. This model has also been used to model the thermal expansion coefficient of composites<sup>5</sup> for which it has been found<sup>6</sup> to provide an accurate description of the experimental data for inclusion contents of up to  $\approx 40$  vol%.

The synthesis and use of uniformly coated (i.e., composite) powders for diverse applications such as pigments, paints, magnetic tapes, drugs and pharmaceuticals, catalysts and dispersion-strengthened alloys have been reviewed by Sparks<sup>7</sup> and Garg and Matijevic.<sup>8</sup> Garg and De Jonghe<sup>9</sup> have discussed the increasing use of coated powders in two major areas of materials processing: (i) the homogeneous incorpo-

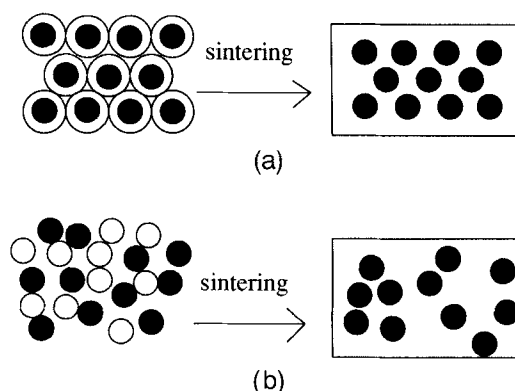


Fig. 1. Schematic diagrams of the compacted composites formed from (a) uniformly coated inclusion particles and (b) conventional mixing of the matrix and inclusion powders.

M. P. Harmer—contributing editor

Manuscript No. 196422. Received August 27, 1991; approved April 9, 1992. Presented at the 93rd Annual Meeting of the American Ceramic Society, Cincinnati, OH, May 2, 1991 (Basic Science Division, Paper No. 164-B-91). Supported by the U.S. Air Force Office of Scientific Research under Grant No. 90-0267.

\*Member, American Ceramic Society.

ration of additives for sintering the microstructure control and (ii) the formation of particulate- or whisker-reinforced materials. In these two areas, procedures have been developed for the preparation of uniformly coated inorganic particles by forced hydrolysis of metal salt solutions in the presence of the core particles, including hematite on titania,<sup>10</sup> aluminum hydrous oxide on hematite, chromia, and titania,<sup>11,12</sup> yttria or alumina–yttria on silicon nitride whiskers,<sup>9</sup> and alumina on silicon carbide whiskers.<sup>13</sup> Sacks *et al.*<sup>14</sup> have used the controlled hydrolysis of tetraethyl orthosilicate to synthesize composite powders of alumina coated with amorphous silica.

The present work reports on the preparation and sintering of composite powders consisting of an inert, particulate core of  $\text{ZrO}_2$  (i.e., the inclusions) and a thick coating of ZnO (i.e., the matrix phase of the composite body). The ZnO was precipitated onto the core particles by controlled hydrolysis of zinc chloride solution. Three different inclusion sizes (1, 3, and 14  $\mu\text{m}$ ) were used by selecting the size of the starting  $\text{ZrO}_2$  powder and the volume fraction of the inclusions was controlled by the amount of ZnO precipitated. The  $\text{ZrO}_2$  inclusions were the same as those used in the work described in the preceding paper. The coated powders were compacted by uniaxial pressing in a die and then sintered at a constant heating rate of  $4^\circ\text{C}/\text{min}$  to  $1500^\circ\text{C}$ . The sintering kinetics and microstructural observations were compared with the results described in the preceding paper for similar composites produced by more conventional forming methods.<sup>1</sup>

## II. Experimental Procedure

### (1) Starting Materials

Unstabilized  $\text{ZrO}_2$  powder (Grade SC, Magnesium Elektron Inc, Flemington, NJ) with three different average particle sizes of 1, 3, and 14  $\mu\text{m}$  was used as the core particles. The inclusions were identical to those used earlier<sup>1</sup> in the formation of ZnO matrix composites by more conventional methods. Reagent-grade zinc chloride and urea (Aldrich Chemical Co., Milwaukee, WI) were used for precipitating the ZnO from solution. All solutions and suspensions were prepared in distilled water.

Prior to the coating process, each  $\text{ZrO}_2$  powder was heated at  $1600^\circ\text{C}$  for 5 h to densify any powder agglomerates. The calcined powder was then dispersed in water and the coarse agglomerates were allowed to settle for a few minutes. The supernatant was decanted and the particles were recovered by sedimentation. The sediment was dried at  $\approx 100^\circ\text{C}$  for several hours.

### (2) Coating of the $\text{ZrO}_2$ Inclusion Particles with ZnO

Originally, attempts were made to coat the zirconia particles by the same procedure described by Haile *et al.*<sup>15</sup> for the synthesis of spherical ZnO powders from zinc chloride solution. This procedure resulted in the nucleation and growth of individual particles which did not coat the  $\text{ZrO}_2$  particles. Modification of the procedure to produce a precursor powder by hydrolysis of zinc chloride in aqueous solutions containing urea resulted in coating of the  $\text{ZrO}_2$  particles. This procedure is similar to that described by Garg and De Jonghe<sup>9</sup> for the coating of silicon nitride powder or whiskers with yttria and yttria–alumina precursors.

In the experiments, the concentrations of the inclusion particles and reactants consisted of 2.5 g/L  $\text{ZrO}_2$ , 0.2 mol/L zinc chloride, and 0.7 mol/L urea. The systems were aged at  $75^\circ\text{C}$  with constant stirring for up to 20 h to produce coatings with the desired thickness. After aging, the dispersions were cooled and the solids recovered by centrifuging. The solids were washed with distilled water to remove excess solute and dried at  $150^\circ\text{C}$  for 24 h. The mass of the inclusion particles in the solids was taken to be the initial mass of the  $\text{ZrO}_2$  dispersed in the solution. The thickness of the coatings on the inclusion particles was controlled by the time of aging. In this way,

coated powders containing up to 50 vol% core particles (based on the fully dense composite body) were produced. Inclusion particles with average sizes of 1, 3, and 14  $\mu\text{m}$  were coated in the experiments. In a separate experiment, pure ZnO powder (i.e., containing no  $\text{ZrO}_2$  particles) was precipitated under the identical conditions used for the coated powders.

The pure ZnO powder and the coated powders were characterized by X-ray diffraction, energy dispersive X-ray analysis (EDAX) in the scanning electron microscope, scanning electron microscopy (SEM), and transmission electron microscopy (TEM).

### (3) Sintering of the Coated Powders

The powders were compacted in a uniaxial die to form green bodies (6 mm in diameter by 6 mm) with approximately the same matrix density ( $0.52 \pm 0.02$  of the theoretical density of ZnO). The powder compacts were sintered in flowing dry air ( $50 \text{ cm}^3/\text{min}$ ) at a constant heating rate of  $4^\circ\text{C}/\text{min}$  to  $1500^\circ\text{C}$  in a dilatometer (Dilatronic,  $1600^\circ\text{C}$ ; Theta Industries Inc., Port Washington, NY) that allowed continuous monitoring of the shrinkage kinetics. The density at any temperature was determined from the initial density and the shrinkage. The final densities of selected samples were checked by the Archimedes method. The microstructures of the sintered bodies were observed by SEM of fracture surfaces or polished surfaces.

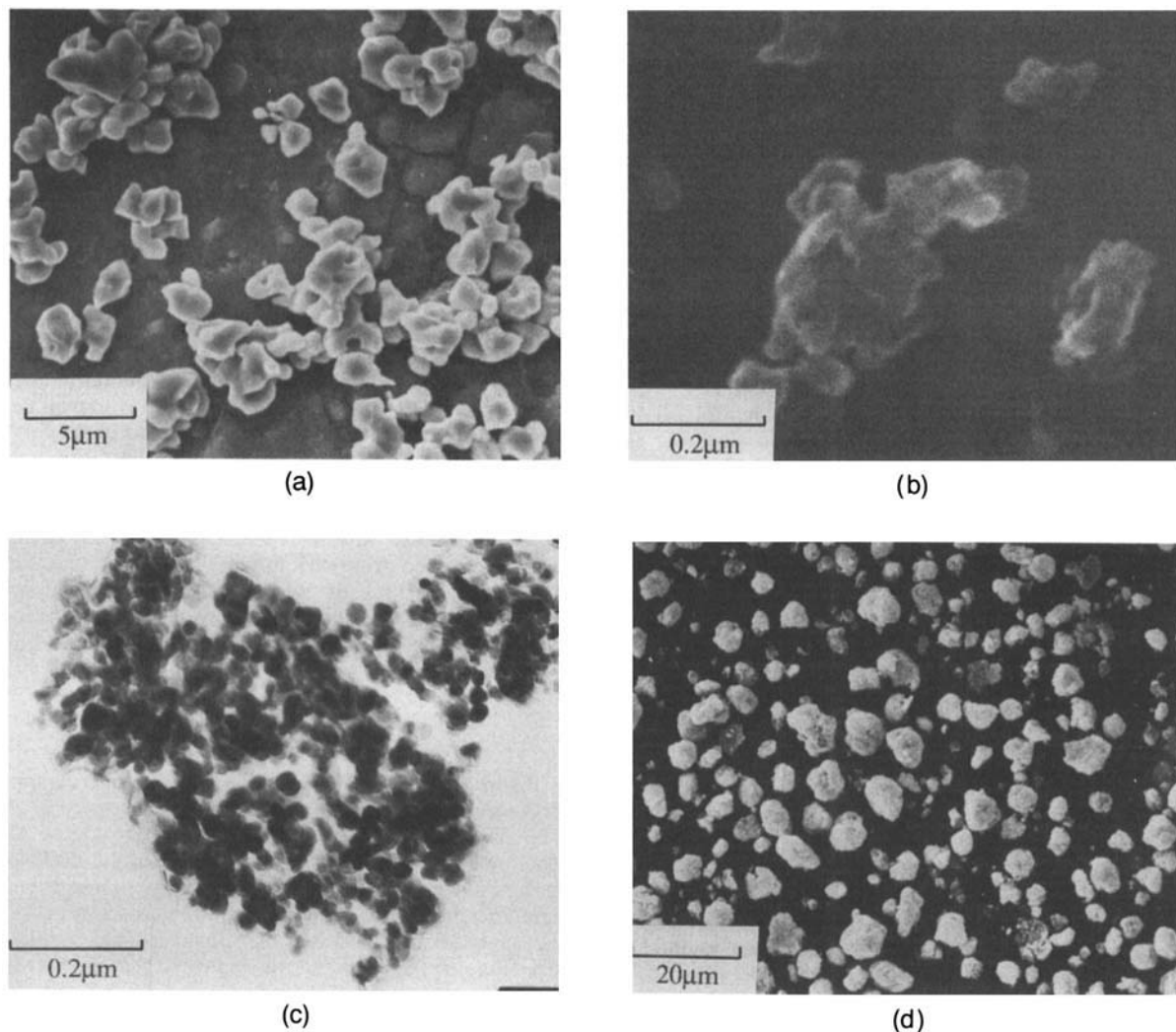
## III. Results

Figure 2(a) shows a scanning electron micrograph of the  $\text{ZrO}_2$  core particles with an average particle size of  $\approx 3 \mu\text{m}$ . The particles are angular and have sharp edges. The pure ZnO powder precipitated in the absence of the  $\text{ZrO}_2$  inclusion particles is shown in Fig. 2(b). The powder appears somewhat agglomerated. Transmission electron microscopy revealed that the agglomerates consisted of primary particles  $\approx 0.1 \mu\text{m}$  in size (Fig. 2(c)). X-ray analysis indicated that ZnO powder was crystalline in nature. The coated powder produced after 18 h of forced hydrolysis is shown in Fig. 2(d). The coated layers appear somewhat rough. Energy dispersive X-ray analysis in the SEM (Fig. 3), as well as X-ray diffraction, confirmed that the  $\text{ZrO}_2$  inclusion particles were coated with the ZnO.

The densification of the compacted powders during constant-heating-rate sintering is shown in Fig. 4, where the composite matrix relative density,  $\rho_m$ , is plotted as a function of temperature,  $T$ , for the pure ZnO and the coated powder containing 10, 20, and 35 vol%  $\text{ZrO}_2$  inclusions (based on the fully dense composite). The average size of the inclusion particles was 3  $\mu\text{m}$ . The data are the average for two runs under identical conditions and each curve is reproducible to  $\pm 0.02$ . The pure ZnO starts sintering at  $\approx 1000^\circ\text{C}$  and reaches a final density of  $\approx 0.92$  at  $1500^\circ\text{C}$ . The composites start sintering at a slightly higher temperature but reach a higher final density (0.97–0.99) at  $1500^\circ\text{C}$ . The higher final density of the composite matrices is somewhat surprising. This might result from slight differences in the characteristics of the ZnO powder precipitated onto the  $\text{ZrO}_2$  surfaces or in the absence of the  $\text{ZrO}_2$ .

An interesting observation from Fig. 4 is that for the range of volume fraction investigated, the densification of the composite bodies is not influenced significantly by the volume fraction of the inclusion particles. The data also indicate that composites with quite an appreciable volume fraction of an inclusion phase can be sintered to nearly full density under identical conditions employed for the unreinforced matrix.

The matrix densification rate of the composite,  $\dot{\epsilon}_m$ , defined as  $(1/\rho_m)(d\rho_m/dt)$ , was calculated by fitting smooth curves to the data of Fig. 4 and differentiating with respect to time,  $t$ . The results for  $\dot{\epsilon}_m$  as a function of temperature,  $T$ , are shown in Fig. 5. Compared to the data for the pure ZnO, the maximum in the curves for the composites shifts to higher  $T$ , and has a higher value. The higher maximum in the curves for



**Fig. 2.** (a) SEM of the  $\text{ZrO}_2$  inclusion particles with an average size of  $3 \mu\text{m}$ , (b) SEM of the pure ZnO powder synthesized by precipitation from solution, (c) TEM of the pure ZnO powder, and (d) SEM of the coated inclusion particles after 18 h of precipitation under the conditions described in the text.

the composites is somewhat surprising. As outlined earlier for the density data, this might arise from slight differences in the ZnO powder characteristics. However, the data for the three composites fall within a narrow band and this is an indication of the weak influence of the inclusion volume fraction on the sinterability of the composites.

In Fig. 6, the sinterability of a composite formed from the coated inclusions particles is compared with that for a body formed more conventionally by mixing, in a ball-mill, the inclusion particles with the pure ZnO powder. (The pure ZnO powder was synthesized under identical conditions used for the coated powders.) The two composites contain an equivalent inclusion content (20 vol%) of  $\text{ZrO}_2$  particles with the same inclusion size ( $3 \mu\text{m}$ ). The data show that the composite formed from the physically mixed powder has a significantly lower sinterability compared with the composite formed from the coated inclusion particles.

Figure 7 shows the effect of the  $\text{ZrO}_2$  inclusion size on the densification of the composites formed from the coated powders, for a fixed inclusion content of 20 vol% (based on the fully dense composite). For the range of core particle sizes used (1, 3, and  $14 \mu\text{m}$ ), the sintering kinetics are almost independent of the inclusion size.

A scanning electron micrograph of the polished surface of a sintered composite formed from the coated inclusion particles is shown in Fig. 8(a). The sample was sintered at  $4^\circ\text{C}/\text{min}$  to  $1500^\circ\text{C}$ , and the inclusion content was 20 vol%. The sam-

ple appears almost fully dense. The porosity and damage, especially in the matrix regions immediately surrounding the inclusions, which were observed for the sintered composites formed by more conventional methods (see Ref. 1, Fig. 10), are absent in these composites. Figure 8(b) is a scanning electron micrograph of the fracture surface of the composite shown in Fig. 8(a). It reveals a polycrystalline matrix with an average grain size of  $\approx 5 \mu\text{m}$ .

#### IV. Discussion

In the preparation of coated powders by precipitation from solution,<sup>8,9</sup> a number of interactions are possible between the suspension of core particles and the material being released from solution. In the present study, the nature of the interactions between the  $\text{ZrO}_2$  inclusion particles and the precipitated ZnO powder is, as yet, unclear, and is currently under investigation. However, the scanning electron micrographs (Fig. 2) and the X-ray analysis data (Fig. 3) indicate that the  $\text{ZrO}_2$  inclusion particles are indeed coated with the ZnO powder. The main focus of the present discussion is the sintering characteristics of these coated powders and how they compare with the data obtained earlier<sup>1</sup> for composites formed more conventionally, from mixtures of  $\text{ZrO}_2$  and ZnO powders.

The data of Fig. 4 show that the sinterability of the composites formed from the coated powders is only weakly dependent on the volume fraction of the  $\text{ZrO}_2$  and that composites

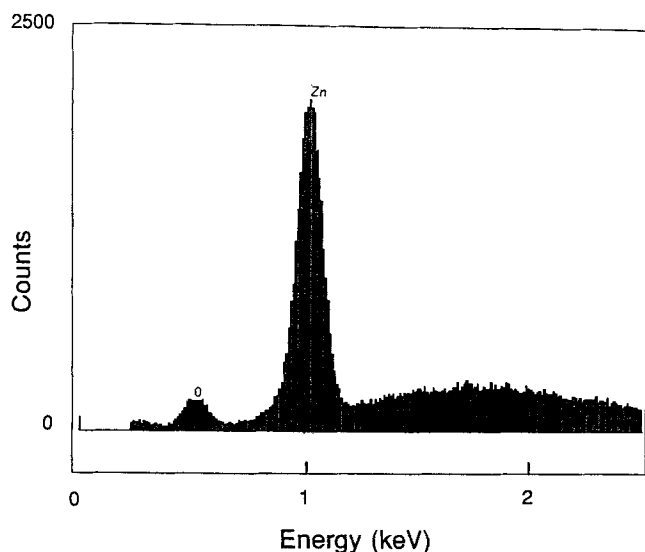


Fig. 3. Energy-dispersive X-ray analysis of the coated  $\text{ZrO}_2$  inclusion particles, showing no Zr peaks.

containing up to  $\approx 40$  vol%  $\text{ZrO}_2$  can be sintered to full density. This is a remarkable improvement over polycrystalline composites formed more conventionally by mixing of the matrix and inclusion powders where modest inclusion contents of  $< \approx 10$  vol% have been found to lead to a severe reduction in the sinterability of the composite (see the preceding paper<sup>1</sup>).

Figure 4 also shows that under identical sintering conditions ( $4^\circ\text{C}/\text{min}$  to  $1500^\circ\text{C}$ ), the pure ZnO (i.e., precipitated in the absence of  $\text{ZrO}_2$  core particles) reaches a lower final density ( $\approx 0.92$ ) compared to the composite matrix even though it starts sintering at a slightly lower temperature. Moreover, the final density of the composite matrix improves with increasing volume fraction of  $\text{ZrO}_2$  inclusion particles up to  $\approx 20$  vol%, above which nearly full density is reached. For identical packing of the same matrix, the sintered matrix density of composites containing coarse, inert inclusions should not increase with increasing inclusion content. Normally, there is a considerable reduction, as shown earlier.<sup>1</sup> The improvement with increasing inclusion content observed here is not clearly understood but is believed to reflect differences in the coated powders produced by the changing concentration of the inclusion particles. As pointed out by Garg and De Jonghe,<sup>9</sup>

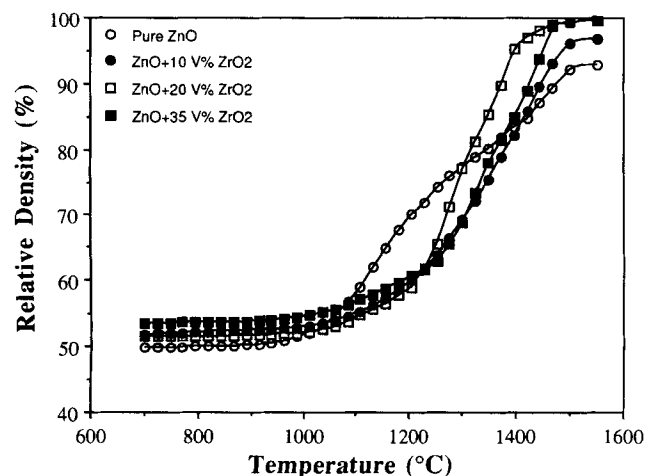


Fig. 4. Matrix relative density vs temperature for the pure ZnO and the composites formed from the coated inclusion particles, during constant-heating-rate sintering at  $4^\circ\text{C}/\text{min}$  to  $1500^\circ\text{C}$ .

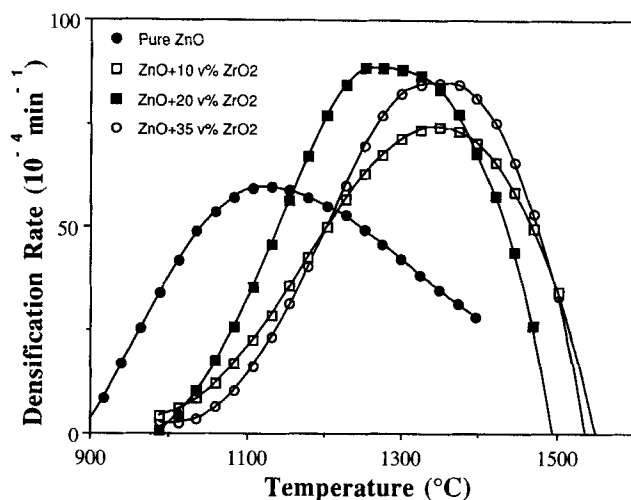


Fig. 5. Densification rate vs temperature for the samples described in Fig. 4.

the surface area of the core particles must be sufficient to prevent the concentration of the reactant from exceeding the critical concentration for homogeneous nucleation, otherwise a mixed system of free precipitates and coated particles will result. Further work is required to understand more clearly how the various synthesis parameters influence the characteristics of the precipitated powder.

It is clear from the results of Fig. 7 that the  $\text{ZrO}_2$  inclusion size, over the range of 1 to  $14\ \mu\text{m}$ , has an insignificant effect on the sinterability of the composites for an inclusion content of 20 vol% used in the present work. This is quite different from earlier observations<sup>1</sup> for conventionally mixed powders where the same  $\text{ZrO}_2$  inclusions produced a significant decrease in the matrix density of the composite with decreasing inclusion size. The absence of an inclusion size effect, as observed in this work, follows from a composite sphere model<sup>5</sup> in which the composite is represented by a collection of composite spheres of various sizes such that each composite sphere contains an inclusion particle and a concentric cladding of matrix material.

The results of the present work show that the sinterability of ceramic-matrix composites is a processing-related problem. They provide conclusive support for the suggestion made in the previous paper<sup>1</sup> that the important factors which control

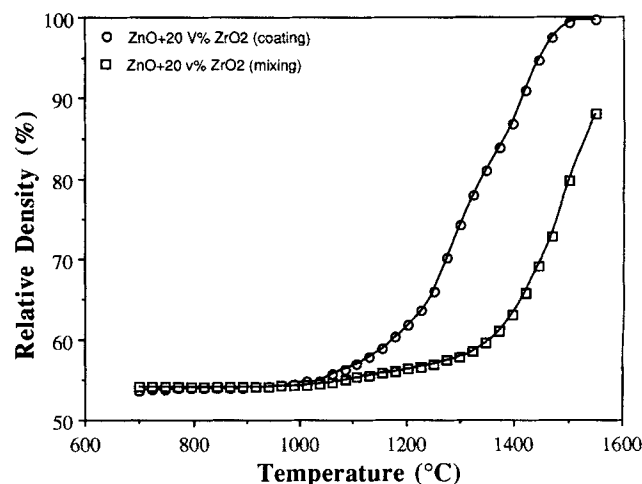


Fig. 6. Matrix relative density vs temperature for the composite formed from the coated inclusion particles (20 vol% inclusions), compared with the data for a composite formed from conventionally mixed powders.

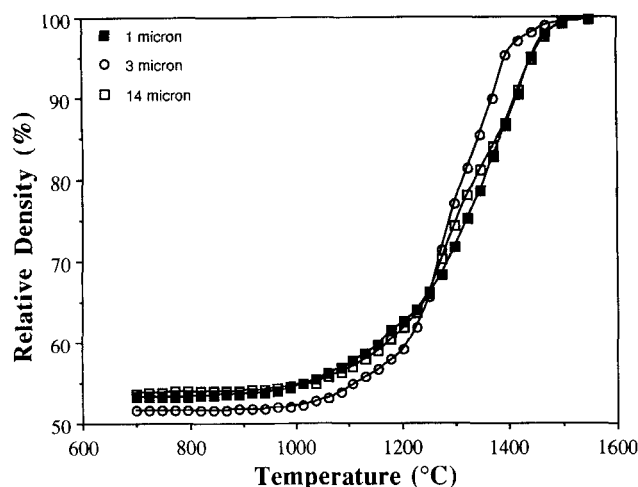


Fig. 7. Effect of the  $\text{ZrO}_2$  inclusion particle size on the sinterability of the composites formed from the coated powders for a  $\text{ZrO}_2$  content of 20 vol%.

the sintering of ceramic composites are (i) inclusion–inclusion interactions which constrain the matrix and (ii) the packing of the matrix phase, especially in the regions immediately surrounding the inclusions. When these factors are minimized or avoided, as in the present work with coated inclusion par-

ticles, then composites containing high inclusion contents can be conventionally sintered, by solid-state diffusion processes, to nearly full density under the same processing conditions used for the unreinforced matrix.

## V. Conclusions

The use of coated powders for the formation of polycrystalline  $\text{ZnO}$  matrix composites containing  $\text{ZrO}_2$  particulate inclusions resulted in a remarkable improvement in the sinterability of the composites. Composites containing up to  $\approx 40$  vol% inclusions were sintered to nearly full density during constant heating rate sintering at  $4^\circ\text{C}/\text{min}$  to  $1500^\circ\text{C}$ .

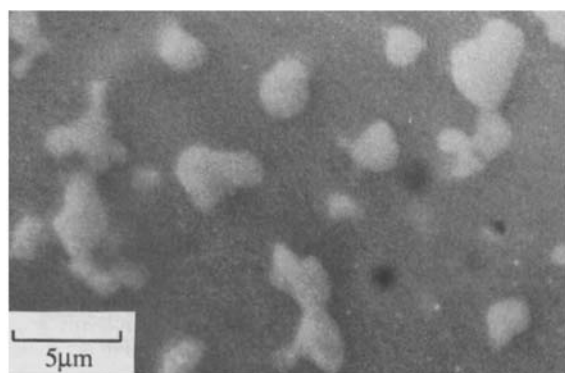
For inclusion contents up to 40 vol%, the matrix sinterability of the composites showed no significant dependence on the volume fraction of the inclusion phase. The zirconia inclusion size, over the range of 1 to  $14\ \mu\text{m}$  used in this work, has an insignificant effect on the sinterability of the composites. These observations are quite different from earlier data for similar composites formed by more conventional methods where strong inclusion volume fraction and inclusion size effects were observed.

The kinetic data and microstructural observations are consistent with the suggestion put forward in the preceding paper that the main factors controlling the sinterability of ceramic-matrix composites are (i) particle–particle interactions between the inclusion which provide a constraint on the matrix and (ii) the packing of the matrix, especially in regions immediately surrounding the inclusions.

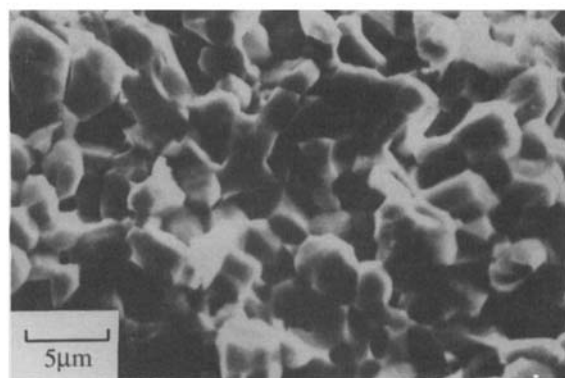
**Acknowledgment:** We would like to thank Professor Lutgard C. De Jonghe for his valuable comments and suggestions.

## References

- <sup>1</sup>C.-L. Fan and M.N. Rahaman, "Factors Controlling the Sintering of Ceramic Particulate Composites: I, Conventional Processing," *J. Am. Ceram. Soc.*, **75** [8] 2056–65 (1992).
- <sup>2</sup>C. H. Hsueh, A. G. Evans, R. M. Cannon, and R. J. Brook, "Viscoelastic Stresses and Sintering Damage in Heterogeneous Powder Compacts," *Acta Metall.*, **34** [5] 927–36 (1986).
- <sup>3</sup>R. Raj and R. K. Bordia, "Sintering of Bimodal Powder Compacts," *Acta Metall.*, **32** [7] 1003–19 (1984).
- <sup>4</sup>G. W. Scherer, "Sintering with Rigid Inclusions," *J. Am. Ceram. Soc.*, **70** [10] 719–25 (1987).
- <sup>5</sup>T. T. Wang and T. K. Kwei, "Effect of Induced Thermal Stresses on the Coefficients of Thermal Expansion and Densities of Filled Polymers," *J. Polym. Sci.*, **7** [A2] 889–96 (1969).
- <sup>6</sup>S. J. Feltham, B. Yates, and R. J. Martin, "The Thermal Expansion of Particulate-Reinforced Composites," *J. Mater. Sci.*, **17**, 2309–23 (1982).
- <sup>7</sup>R. E. Sparks, "Microencapsulation"; p. 470 in *Encyclopedia of Chemical Technology*, Vol. 15. Edited by M. Grayson and D. Eckroth. Wiley, New York, 1981.
- <sup>8</sup>A. Garg and E. Matijevic, "Preparation and Properties of Uniformly Coated Inorganic Colloidal Particles. 2. Chromium Hydroxide on Hematite," *Langmuir*, **4**, 38–44 (1988).
- <sup>9</sup>A. K. Garg and L. C. De Jonghe, "Microencapsulation of Silicon Nitride Particles with Yttria and Yttria–Alumina Precursors," *J. Mater. Res.*, **5** [1] 136–42 (1990).
- <sup>10</sup>P. Gherardi and E. Matijevic, "Interactions of Precipitated Hematite with Preformed Colloidal Titania Dispersions," *J. Colloid Interface Sci.*, **109** [1] 57–68 (1986).
- <sup>11</sup>S. Kratochvil and E. Matijevic, "Preparation and Properties of Coated, Uniform, Inorganic Colloidal Particles: 1, Aluminum (Hydroxide) Oxide on Hematite, Chromia, and Titania," *Adv. Ceram. Mater.*, **2** [4] 798–803 (1987).
- <sup>12</sup>H. Okamura, E. A. Barringer, and H. K. Bowne, "Preparation and Sintering of Monosized  $\text{Al}_2\text{O}_3$ – $\text{TiO}_2$  Composite Powder," *J. Am. Ceram. Soc.*, **69** [2] C-22–C-24 (1986).
- <sup>13</sup>D. Kopolnek and L. C. De Jonghe, "Particulate Composites from Coated Powders," *J. Eur. Ceram. Soc.*, **7** [6] 345–51 (1991).
- <sup>14</sup>M. D. Sacks, N. Bozkurt, and G. W. Scheiffele, "Fabrication of Mullite and Mullite–Matrix Composites by Transient Viscous Sintering of Composite Powders," *J. Am. Ceram. Soc.*, **74** [10] 2428–37 (1991).
- <sup>15</sup>S. M. Haile, D. W. Johnson, G. H. Wiseman, and H. Kent Bowen, "Aqueous Precipitation of Spherical Zinc Oxide Powders for Varistor Applications," *J. Am. Ceram. Soc.*, **72** [10] 2004–2008 (1989).



(a)



(b)

Fig. 8. SEM of (a) a polished surface and (b) a fracture surface of the composite formed from the coated inclusion particles after sintering at  $4^\circ\text{C}/\text{min}$  to  $1500^\circ\text{C}$ . The inclusion volume fraction and size are 20 vol% and  $3\ \mu\text{m}$ , respectively.