DENSIFICATION BEHAVIOR OF SLS PROCESSED Al_2O_3/Al COMPOSITE

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ABSTRACT

Production of structurally sound parts by any rapid prototype technique is essential, because fully functional features are necessary where application testing is required. In the present work, a powder blend of Al_2O_3/Al (3:1 by weight) was mixed with ammonium dihydrogen phosphate and subjected to selective laser sintering (SLS) using a CO_2 laser. An attempt has been made to increase the powder bed density by introducing vibration to the part cylinder. These SLS processed preforms were then subjected to a secondary heat treatment in a hydrogen atmosphere and to hot isostatic pressing. Densification behavior of these Al_2O_3/Al composite preforms is discussed.

INTRODUCTION

Rapid technological developments are driving increased global competition for compressed design and manufacture of new products. In recent years, a number of solid freeform fabrication (SFF) techniques have been developed to produce rapid prototype solid objects. These SFF techniques reduce time and cost of production substantially compared to conventional methods of fabrication of prototype solid parts. However, parts produced by many of these SFF techniques exhibit inferior mechanical properties relative to conventionally produced counterparts. The selective laser sintering (SLS) process, one of the SFF techniques, has commercial feasibility for the production of solid parts using polymeric and wax materials. Presently, the SLS process is in the developmental stage relative to production of metallurgically sound and fully functional parts from metals, ceramics and composite materials [1-6]. Functional features in the prototype solid part are essential where applications testing is required. Successful processing of these materials, however, depends on process parameters described elsewhere [7]. The aim of the present investigation is to study densification behavior of an Al_2O_3/Al composite system under the following conditions: a) introduction of vibration to the part cylinder to increase powder bed density before SLS, b) secondary heat treatment of the SLS processed preforms in hydrogen and c) hot isostatic pressing (HIP) of the SLS processed preforms.

EXPERIMENTAL DETAILS

Electronics grade calcined alumina powder and aluminum powder used in the present study were uniformly mixed in the weight ratio of 3:1, respectively. To this powder blend, 20% by

weight ammonium dihydrogen phosphate, $NH_4H_2PO_4$, was added as a binder. The characteristics of these powders are highlighted in the Table. After proper blending, the composite mix was subjected to SLS using a CO_2 laser. The process parameters maintained during SLS were as follows:

Incident Power	= 14W	;	Scan Speed	= 6.35 cm/s
Beam Diameter	= 0.5 mm	;	Scan Line Spacing	= 5 mils (125 μm)

TABLE: Characteristics of the powders used in this study

Material	Average particle size (μm)	Shape	Purity (%)	Source
Alumina	15	Flake	99.55	Norton Company
Aluminum	20	Spherical	99.00	Johnson Matthey Catalog Company
Ammonium dihydrogen phosphate*	‹ 20	Flake	99.95	Johnson Matthey Catalog Company

* received in crystalline form and ground to ‹ 20 μm size

To study the effect of powder bed density on the SLS processed preforms, the powder bed density was varied before SLS. A stainless steel disc weighing 300 gm was placed on the powder bed surface and with an electric engraver on top of it, and the powder bed was vibrated for a period of 0 to 30 seconds to generate a wide range of powder bed density. To remove the binder from the composite system and to study the densification behavior, these SLS processed preforms (with an average relative density of 46%) were subjected to a secondary heat treatment in a hydrogen atmosphere at two different temperatures, 800°C and 850°C. Soaking time was varied from 2 to 10 hours and the preforms were furnace cooled. Volumetric shrinkage was calculated by measuring the change in dimensions of the preforms after the heat treatment. An attempt was also made to study the densification behavior of the SLS processed preforms during HIP. Initially, SLS processed preforms were encapsulated in pyrex glass before HIP. The temperature, pressure and duration of HIP were 940±10°C, 10,000 psi and 60 minutes. Density was calculated by dividing the mass of the samples with nominal dimensions of 10.0mm X 10.0mm X 3.0mm (± 0.3mm variation) by their true volume determined from linear dimensions and relative density was calculated by dividing the actual density of the SLS processed preforms by the theoretical density of the composite system.

RESULTS AND DISCUSSION

It is known that once the powder bed is tapped or vibrated, there is a change in packing configuration of the powder bed from loose random packing towards close random packing. This results in improvement in the packing density. An attempt was made to study the densification behavior of the SLS processed preforms with the powder bed density. Figure 1 shows the effect of vibration to the $Al_2O_3/Al + NH_4H_2PO_4$ composite powder bed. Curve a represents the powder bed and Curve b represents the SLS processed preform. The relative density of the powder mix in the loose random configuration is 35% and increases up to a maximum of 42% as vibration time is increased. No further improvement in packing density was observed beyond

25 seconds of vibration time. When the powder bed was subjected to SLS after vibration, improvement in the relative density of the SLS preform was seen. This indicates that there is a possibility for improving the initial density of the SLS processed preform by as much as 27% by vibrating the powder bed. Figures 2a and 2b are scanning electron micrographs of the SLS processed Al_2O_3/Al composite preform with 46% relative density. These demonstrate the wettability of the ammonium dihydrogen phosphate. Figure 2a represents a low density region and figure 2b represents a high density region. In both micrographs, it is clear that the Al_2O_3/Al particles are completely wet and covered by the ammonium dihydrogen phosphate. Despite good wetting, part overall density remained less than 50%.

Though ammonium dihydrogen phosphate was used as a binder, like most binders, its presence in the system reduces high-temperature properties of the composite. To remove this phase from the composite system, SLS processed preforms were subjected to a heat treatment in a hydrogen atmosphere. After the heat treatment, in spite of volumetric shrinkage of the SLS processed preforms, the overall density of the preforms was not improved, and in some cases, density actually decreased. This is attributed to the loss of mass due to the evolution of NH_3 and H_2O during the heat treatment. Figure 3 shows the effect of the heat treatment temperature and time on the percent volumetric shrinkage. Curve a represents 800°C and Curve b represents 850°C. Volumetric shrinkage increases with increasing temperature and holding time. This increase in the volumetric shrinkage is higher at initial stages of holding time and tends toward saturation after 8 hrs holding time. Figure 4 is a scanning electron micrograph (cross section) of the heat treated (850°C for 8 hrs) SLS processed preform. The preform has extended interconnected porosity, and the glassy phase enveloping the Al_2O_3/Al particles as shown in figure 2b is removed. However, an earlier study of this binder in SLS [8] indicated that a residue of $AlPO_4$ remains in the system.

Figure 5 is a scanning electron micrograph of a HIP SLS processed preform with 76% theoretical density. In spite of the high temperature and pressure used during HIP, full density could not be achieved for this system, and interconnected porosity is still present. Generation of ammonia and water vapor in the pores may have contributed to incomplete closure of pores.

CONCLUSIONS

The salient features of the present study are : a) improvement in initial density of the SLS processed preforms up to 27% is observed by increasing the powder bed density via vibration of the part cylinder; b) by heat treating the SLS processed preforms, the glassy phase in the composite system could be removed but with concomitant loss of density ; and c) maximum relative density that could be achieved for this system by HIP is approximately 76%.

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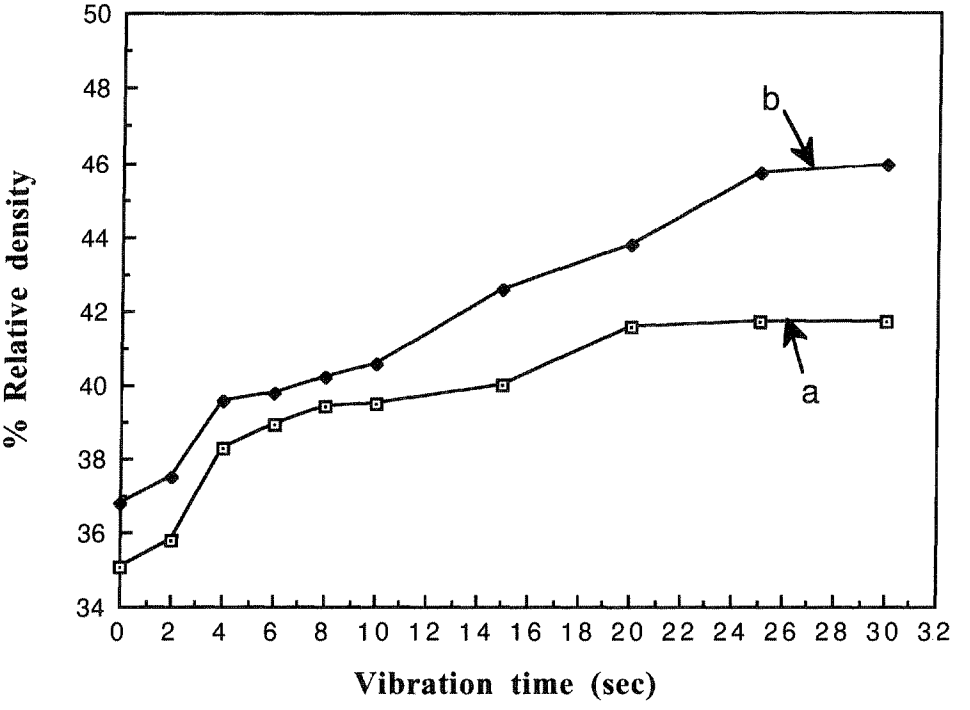

Fig.1 Effect of vibration on percent relative density of the composite a) powder bed and b) SLS processed preform

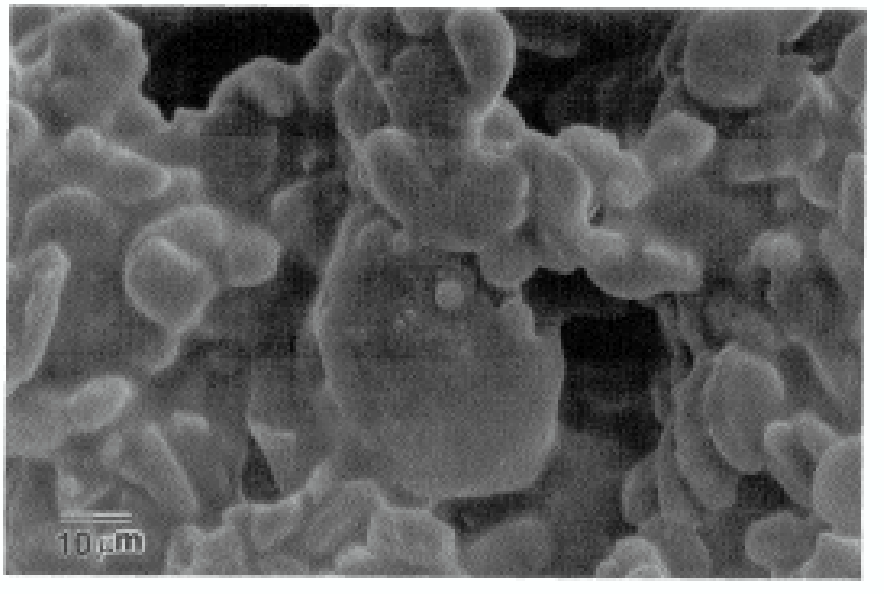

Fig. 2a SEM micrograph of the Al_2O_3/Al composite at a low density region (30 seconds vibration, 46% relative density)

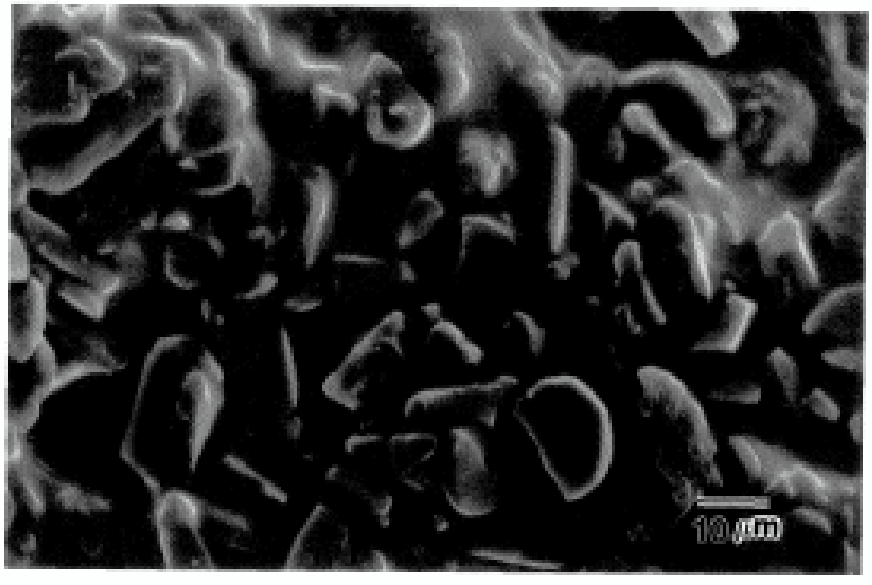

Fig.2b SEM micrograph of the Al_2O_3/Al composite at a high density region (30 seconds vibration, 46% relative density)

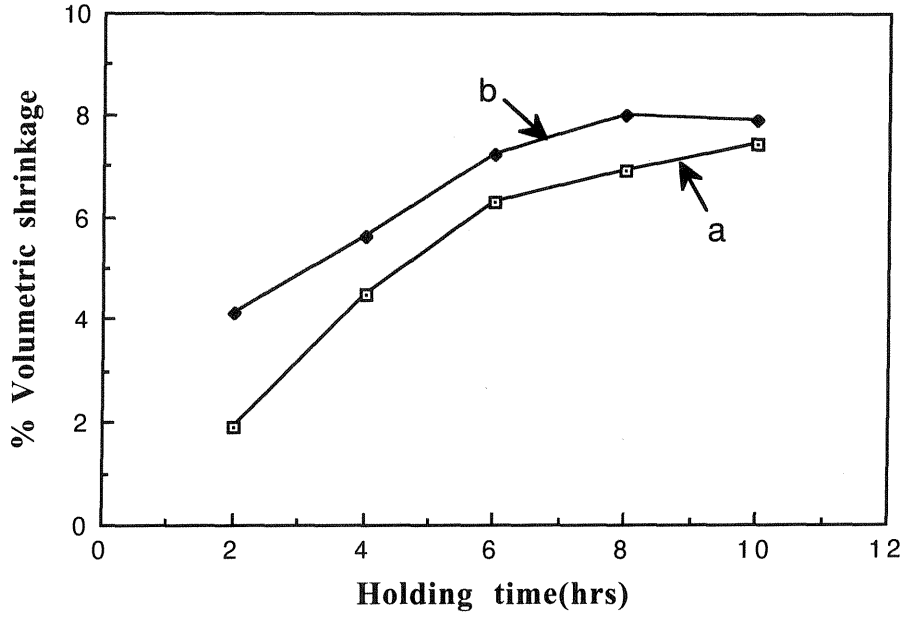

Fig.3 Effect of post-SLS heat treatment temperature and time on percent volumetric shrinkage ; a) 800°C and b) 850°C, Hydrogen atmosphere

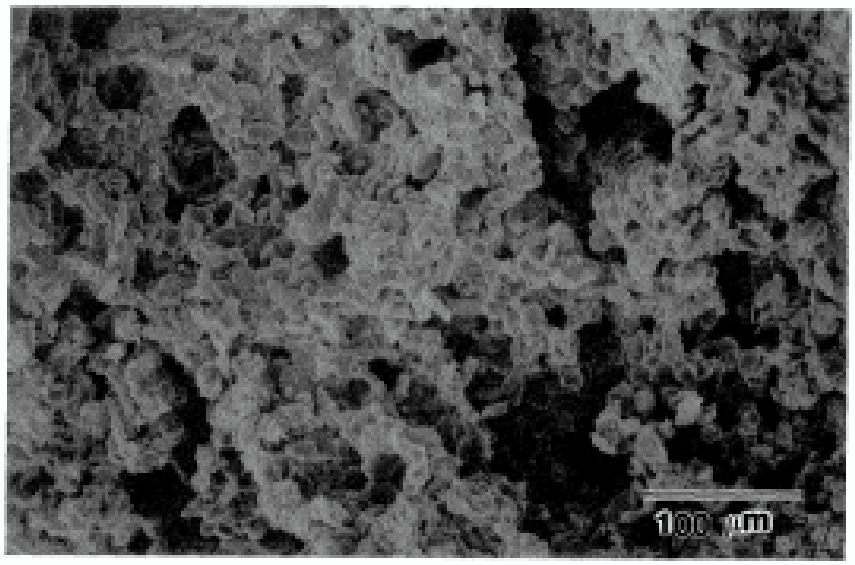

Fig.4 SEM micrograph of the heat treated SLS processed preform (30 seconds vibration, 46% relative density, HT at 850°C for 8 hr, Hydrogen atmosphere)

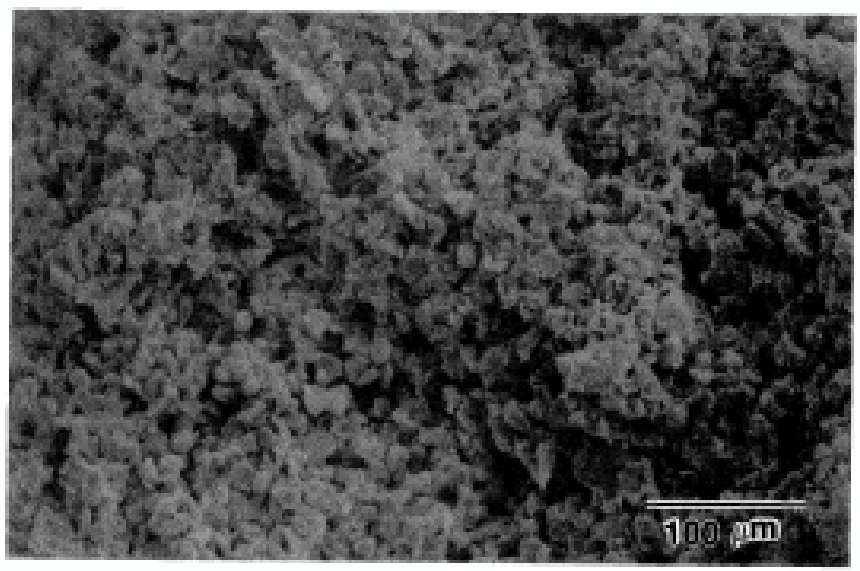

Fig.5 SEM micrograph of the HIP SLS processed preform (30 seconds vibration, 46% relative density, HIP at 940±10°C for 60 minutes)