Selective Laser Sintering of Al_2O_3.

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Abstract

Selective Laser Sintering (SLS) of Al_2O_3 with an organic binder to create the green part is reported. The effect of processing parameters on the strength and density of parts produced by SLS are examined. The effect of particle size on the process and resulting green strength was studied. Two methods of applying the polymer binder to the ceramic powder, i) spray drying and ii) mixing, are compared.

Introduction

Selective Laser Sintering (SLS) has been employed as a method of forming parts from Al_2O_3. Three approaches to SLS of Al_2O_3 may be employed to make parts. These are i) use an inorganic binder[1,2,3] to bond the Al_2O_3 particles, ii) use an organic binder such as poly methyl methacrylate[4] (PMMA) to bond the alumina and iii) direct sintering of the Al_2O_3 powder. This paper describes the experiments done on the laser sintering of Al_2O_3 powder of various sizes with organic binders and reports the properties of the green SLS parts.

When an organic binder is used the binder may be removed completely at a later stage by burnout. Thus, with the proper choice of the binder, it has the advantage of little material contamination and of being a low temperature SLS process.

Experiments

Three kinds of Al_2O_3 powder were investigated for this study. The characteristics are shown in table I. The 2μm and 8μm powders contained some flocculants that were removed by heating to 800°C.

Agglomeration of the particles as received was observed only for 8μm particles. The 2μm particles had limited agglomeration following the 800C thermal treatment.

Table I

Sample Ave. Particle size	Composition	Agglomeration
2 μm	99.5% Al_2O_3	Some
8 μm	85%Al_2O_3, 2%CaO 1%MgO, Bal SiO_2	Particles were in 70-100μm agglomerates.
15 μm	99.5% Al_2O_3	None

Two methods of applying the PMMA to the powder were used: i) mixing the polymer powder with the ceramic powder and ii)Spray drying the ceramic powder with the polymer emulsion using conditions shown in table II. The polymer powder used for the mix was obtained by spray drying the polymer emulsion.

The 2μm powder was spray dried with poly methyl methacrylate (PMMA) emulsion to 35 and 25 vol% respectively according to conditions listed in table II.

Table II Spray drier conditions

Inlet temperature (°C)	Outlet temperature (°C)	Atomizer speed (RPM)
250	90 to 105	30,000

The agglomerates of the 8μm powder were mixed with PMMA powder produced by spray drying and processed by SLS. Three volume fractions 20, 30and 40 vol% of PMMA were employed. These exhibited good green strengths after SLS.

The agglomerates of the 8μm material were also spray dried with PMMA to 30 vol% after adding a viscosity enhancer (Xantham

gum , 0.5%) and tested by SLS. The Xantham gum was necessary to reduce the settling of the agglomerates.

The 70-100μm agglomerates of 8μm particles were broken down using an attritor. This attrited powder was processed both by mixing with PMMA powder and also by spray drying with PMMA.

The 15μm powder was spray dried with PMMA to 30 vol% and the uncoated 15μm ceramic was added to dilute the concentration of PMMA to 25 vol%. Parts were made and density and bend strength determined. As a comparison the 15μm was also mixed with PMMA powder to 25 vol% and parts made by SLS and density and bend strength determined.

The following processing parameters were used for the SLS experiments. The powder bed was maintained at a temperature of about 85°C and laser power kept a constant at 10W. The scan line spacing was fixed at 0.005". The layer thickness was fixed at 0.007". The beam speed was varied to alter the power density incident on the layer. Power density is defined as the amount of energy incident on the layer per unit area in cal/cm^2 . Specimens were tested for both bend strength and density. The volume of the specimen was determined by mensuration to estimate the density. The bend strength was determined with a three point bend test using a INSTRON constant displacement rate machine.

Results and discussion.

SLS of parts from the 2μm powder at both 25 and 35 vol% produced low strength green parts. Strong green parts were produced when the agglomerates of 8μm powder was mixed with PMMA powder and processed by SLS. The variation of bend strength and density with power density of these samples are shown in Figure 1 and Figure 2. When the agglomerated powder was spray dried with PMMA the agglomerates broke down during spray drying. Strong parts were not produced by SLS from the spray dried powder. When the 8μm powder is deagglomerated it does not form

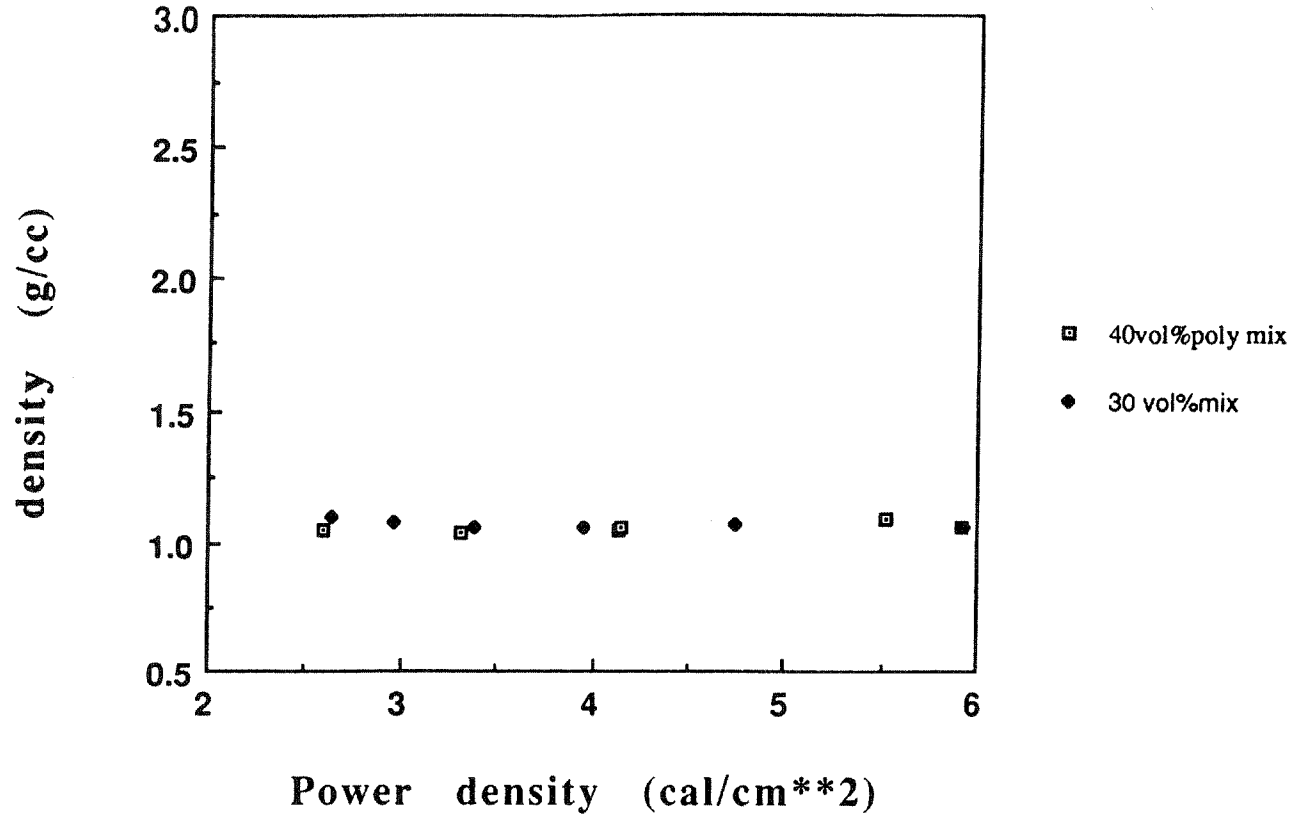


Fig.1. Effect of power density and binder volume fraction on density of parts from agglomerates of 8μm powder by SLS

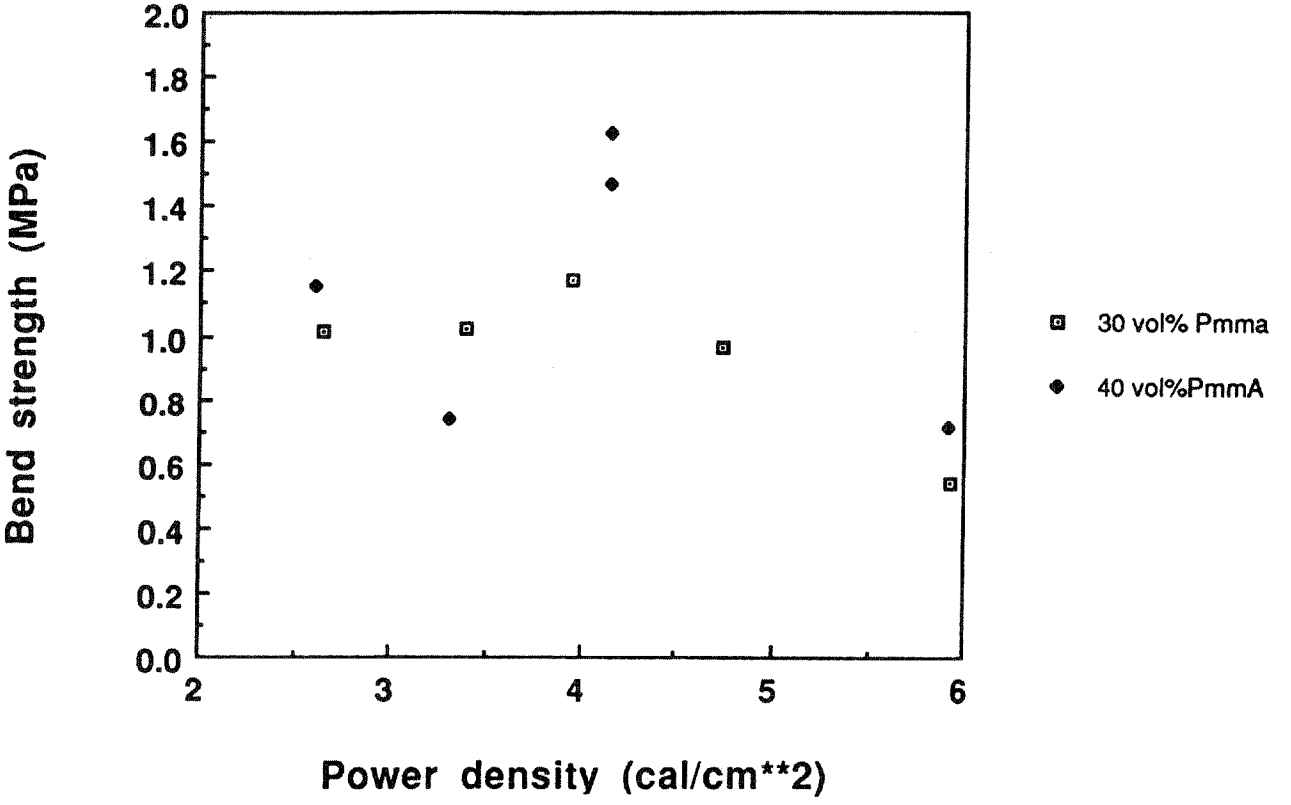


Fig.2 Effect of power density and binder volume fraction on bend strength of parts from agglomerates of 8μm powder by SLS

strong parts when mixed or spray dried with PMMA partly due to the nonuniform coating of the powder. The 15μm powder mixed and spray dried produced reasonable green strengths. The bend strength and density of the samples from 15μm powder are shown in Figure 4 and Figure 5.

Effect of binder content on strength.

Equation 1 relates the effect of binder content to the strength of composite[5].

$$\sigma = k\,(V_b/V_p)^{0.75}\,(\gamma E/G)^{0.5} \qquad (1)$$

where σ =strength of porous material, k= constant, V_b=volume content of binder phase , V_p= volume content of primary phase,

γ= Surface fracture energy, E= Young's modulus and G= Grain size. This equation was derived for brittle materials built up of particles joined at points of contact. A curve fit of the strength to the V_b/V_p ratio for three volume fractions of the polymer in the case of

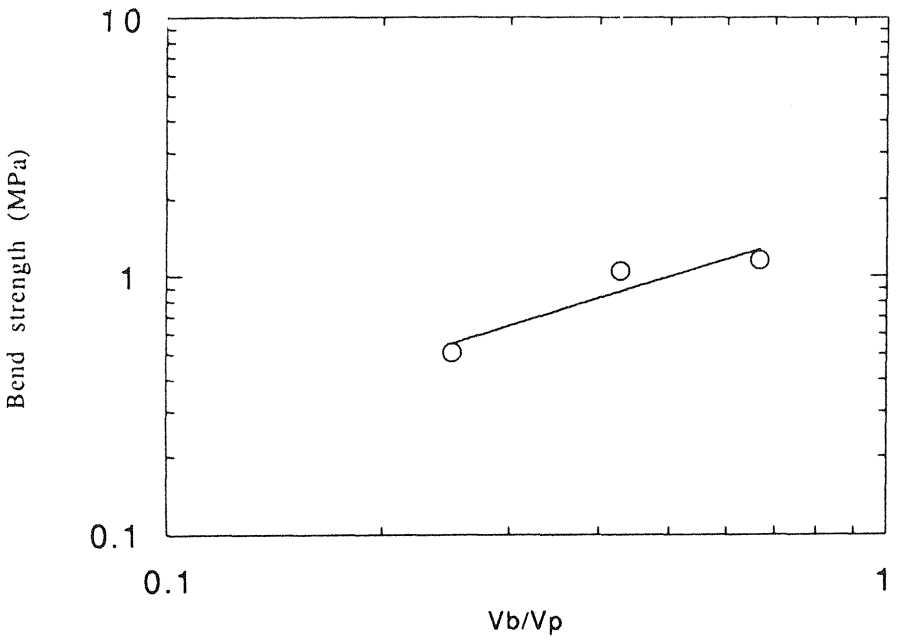


Figure3. Effect of binder volume fraction on bend strength

the agglomerates of 8μm powder is shown in Figure 3. The exponent according to the curve fit is 0.86 with R=0.92. Further experimentation is necessary to establish an appropriate model.

This result that the bend strength will decrease with decreasing content of polymer binder is expected since all the strength is associated with the binder and binder/ceramic interaction.

Effect of binder content on density.

There is not much variation in observed density of the parts with binder content. At a higher volume fraction of polymer there is sufficient polymer to melt and flow and cause an increase in density beyond the powder bed density. At low volume fractions of polymer this is not the case. Hence even though the theoretical density of powder mixtures of 20 and 40 vol% PMMA is 3.41 and 2.86, respectively, the density of parts made from them is approximately the same, 1.05g/cc.

Effect of power density on strength.

At low power densities insufficient melting and flow of binder occurs causing poor strength. At high power densities decomposition of the polymer occurs over a wider area causing a reduction in the strength. An intermediate value of the power density gives a higher strength. This may be seen in Figure 2 and Figure 5.

Effect of particle size on the process.

As the particle size decreases the strength of the part decreases. This is due to the greater surface area that the binder has to contact. If the binder is spread uniformly on all particles this will give rise to a thinner layer of the polymer with decreased particle size. Therefore there will be a smaller bonding area resulting in decreased strength. This may be seen from the fact that both 2μm and 8μm particle sizes do not form strong parts. The 8μm powder when present as agglomerates of 70 - 100 μm size forms strong parts

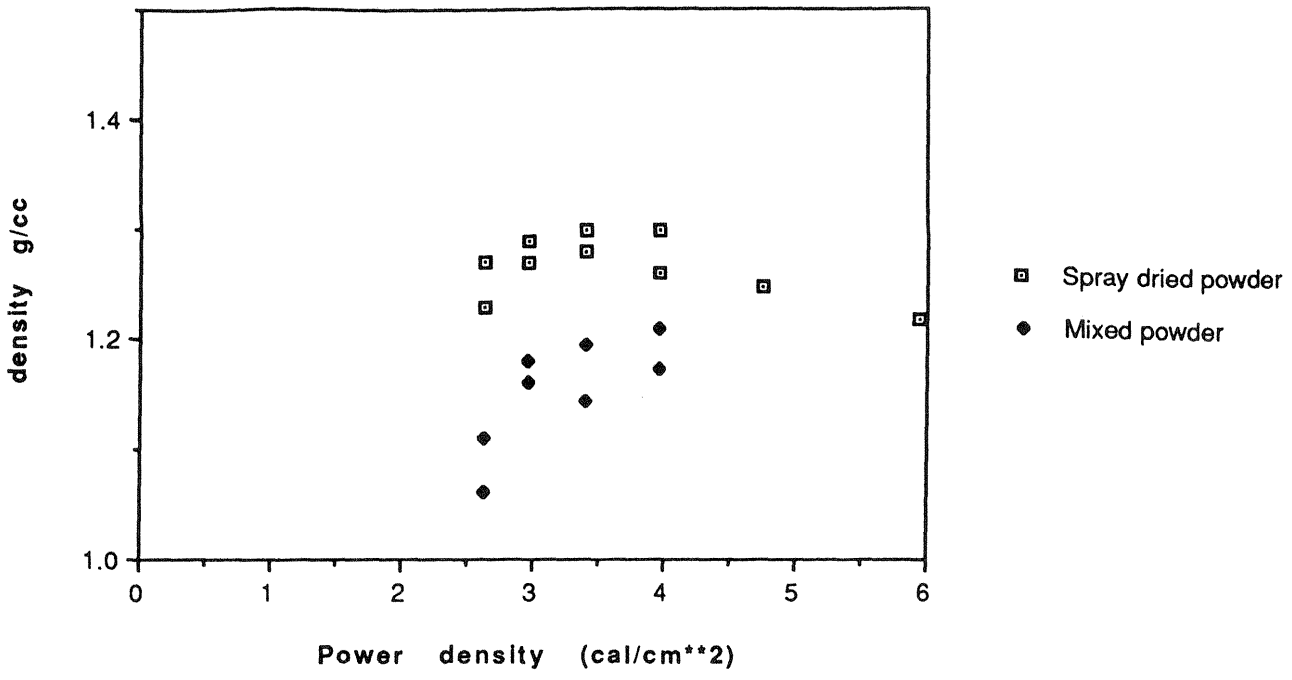


Figure 4. Effect of spray drying .vs. mixing on density of SLS samples for the15μm Al_2O_3.

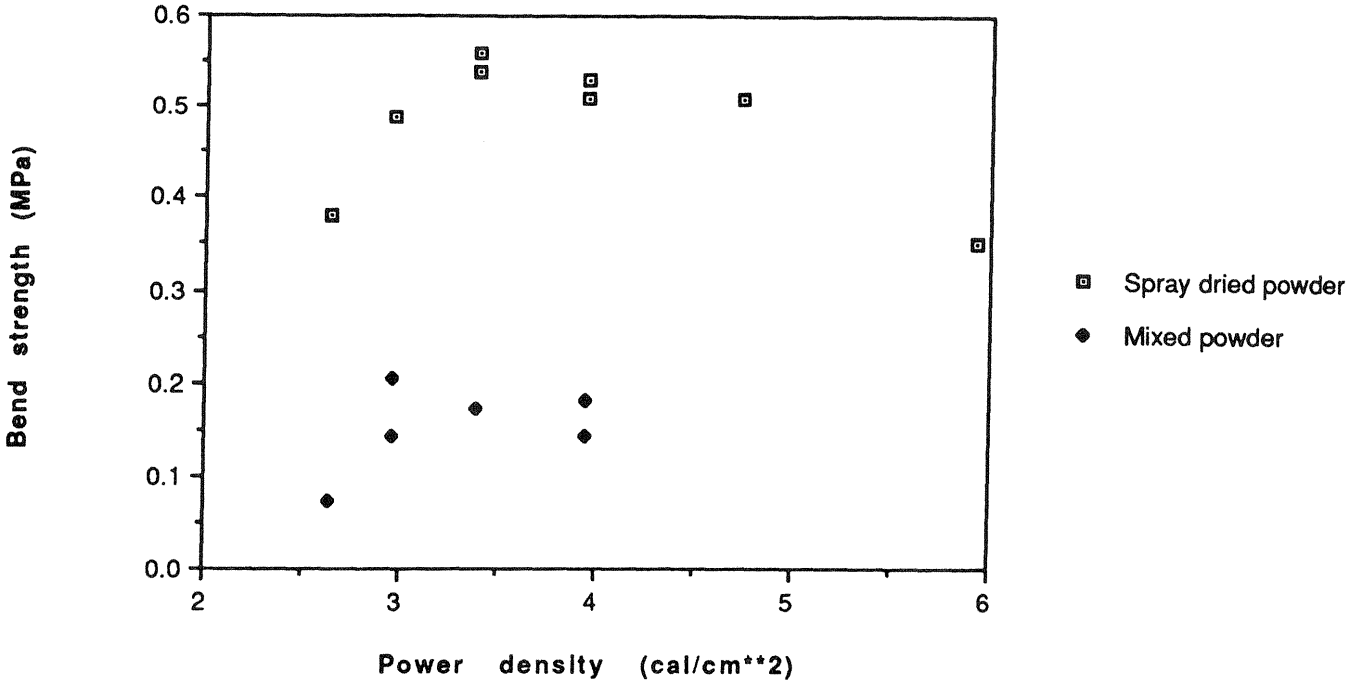
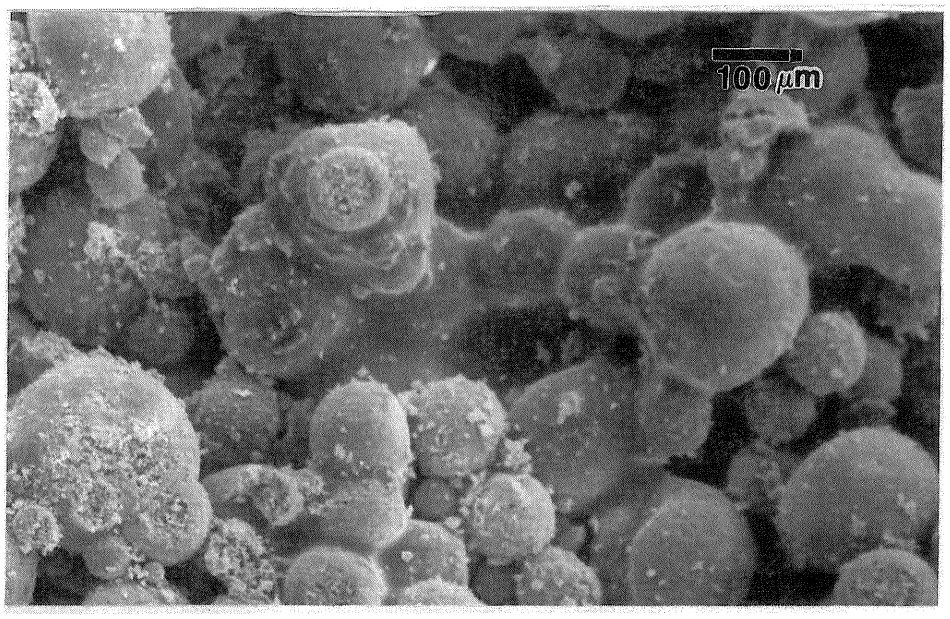
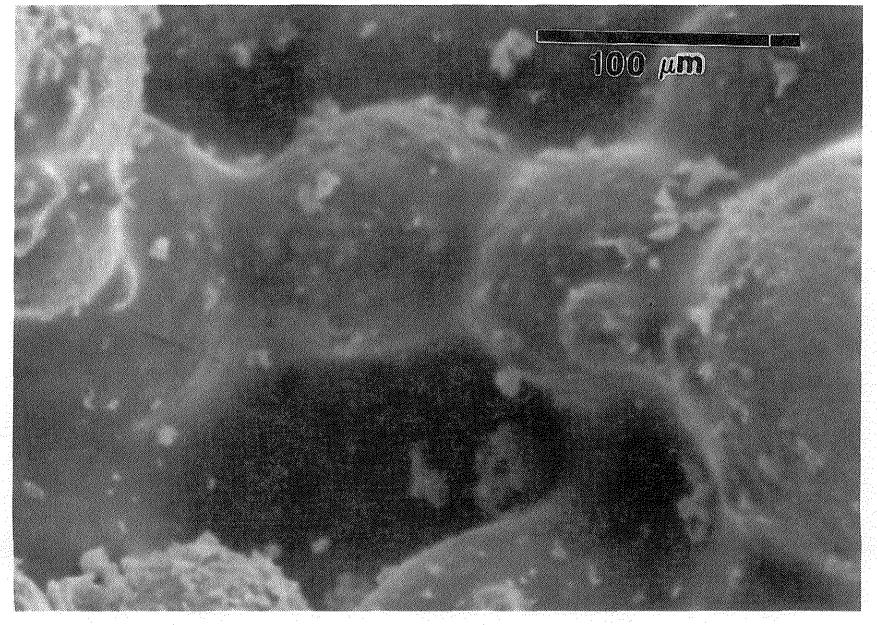


Figure 5. Effect of spraydrying .vs. mixing on bend strength of SLS samples for the 15μm Al_2O_3.



(a)

Figure 6. Microstructure of parts made from agglomerates of 8μm Al_2O_3. a) Low magnification b) High magnification



(b)

when mixed with the polymer. This effect is partially due to the nonuniform coating associated with the spray drying.

Effect of spray drying vs mixing of the polymer.

The effect of spray drying vs mixing for the 15μm Al_2O_3 may be seen in Figure 4 and Figure 5. Under identical conditions the strength for samples from spray dried powder is almost twice the strength for samples from the mixed powder. In the case of spray dried powder the polymer is more homogeneously distributed and the powder more uniformly coated. The 15μm powder is present as 50μm polymer/Al_2O_3 agglomerates in the case of spray dried powder. The density of parts produced by SLS is also higher for spray dried powder as shown in Figure 4.

Microstructure of the composites.

These composites have interconnected porosity with the alumina particles bonded together by the polymer. The polymer envelopes the alumina particles. This may be seen from Figure 6 which shows the surface of a part made by SLS from the agglomerates of 8μm powder.

Summary.

The variables involved in the SLS of alumina using an organic binder are examined. The strength of the green parts produced increases with the volume fraction of the binder. The strength of the parts produced decreases with decreasing particle size. At high laser power density the strength of the parts produced is reduced. Spray drying homogenizes the polymer distribution and strengthens the parts.

References.

1. U. Lakshminarayan and H. L. Marcus, Proceedings of the Solid Freeform Fabriaction symposium, Ed. J.J. Beaman, H.L. Marcus, D.L. Bourell, J.W. Barlow and R.H. Crawford, 1991, p.205

2. U. Lakshminarayan and H. L. Marcus, Proceedings of the Solid Freeform Fabriaction symposium, Ed. J.J. Beaman, H.L. Marcus, D.L. Bourell, J.W. Barlow and R.H. Crawford, 1992, p.44
3. P.K. Subramanian, G. Zong and H.L. Marcus, Proceedings of the Solid Freeform Fabriaction symposium, Ed. J.J. Beaman, H.L. Marcus, D.L. Bourell, J.W. Barlow and R.H. Crawford, 1992, p.63
4. N.K. Vail and J.W. Barlow, Proceedings of the Solid Freeform Fabriaction symposium, Ed. J.J. Beaman, H.L. Marcus, D.L. Bourell, J.W. Barlow and R.H. Crawford, 1992, p.124
5. H.H. Bache, Journal of American Ceramic Society, 53 (12), 1970, p.654

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