

Anisotropy in Alumina Processed by SLS.

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Abstract

Alumina powders of 15 μ m size and 2 μ m size were processed by SLS using PMMA and a copolymer. The 2 μ m powders were agglomerated and mixed with the polymer powder before being processed by SLS. SLS bend strength specimens were made with parts built along different orientations. The variation of the strength with incident energy density and with orientation was studied

Introduction

Selective Laser Sintering (SLS) of ceramics is very challenging because of the high melting points of the ceramics. In SLS one of the operating parameters, the energy density is defined as the amount of laser energy input per unit area¹ in each layer. In the case of alumina to form lines of sintered material energy densities of the order of 1000 cal/cm² is required. Even at this high energy densities the parts formed do not hold together because of the huge thermal gradients. One solution is to use a low melting temperature second phase material which will melt under the laser and bond the particles together. This second phase may be inorganic^{2,3,4} or organic^{5,6}. This paper describes some of the SLS experiments of alumina with organic binder. This paper focuses on the variation of properties of the green shapes with processing parameters including the orientation of scanning in the SLS process.

Experiments

Two kinds of alumina powder were investigated in this study. They were i) 2 μ m powder from Golden Technologies and ii) 15 μ m powder from Norton Corp. SLS of 2 μ m as received powder was unsuccessful using a polymer binder even at compositions of 35 Vol % polymer. In an attempt to overcome this problem it was decided to agglomerate the particles. Through a series of experiments of heating to various temperatures for varied lengths of time it was decided that 1400C for 3hrs would give a desirable agglomeration. The 2 μ m powders were heated in a kiln to 1400C in 12 hrs at a constant heating rate and held at 1400C for 3hrs. The powder was also cooled down in about 12hrs. This produced agglomerates of an average size of 100 μ m. The flow diagram is shown in Fig.1.

The organic binders used were i) PMMA and ii) a copolymer, Poly[methylmethacrylate-co-butyl methacrylate] (80 mol%MMA/20 mol%BMA). The preparation of these polymers is explained elsewhere⁷. The PMMA had a melt index of 31.5g/10 min and the copolymer had a melt index of 11.6g/10min as measured following ASTM D1238 at 200C with an extrusion pressure of 0.52 MPa (75psi).

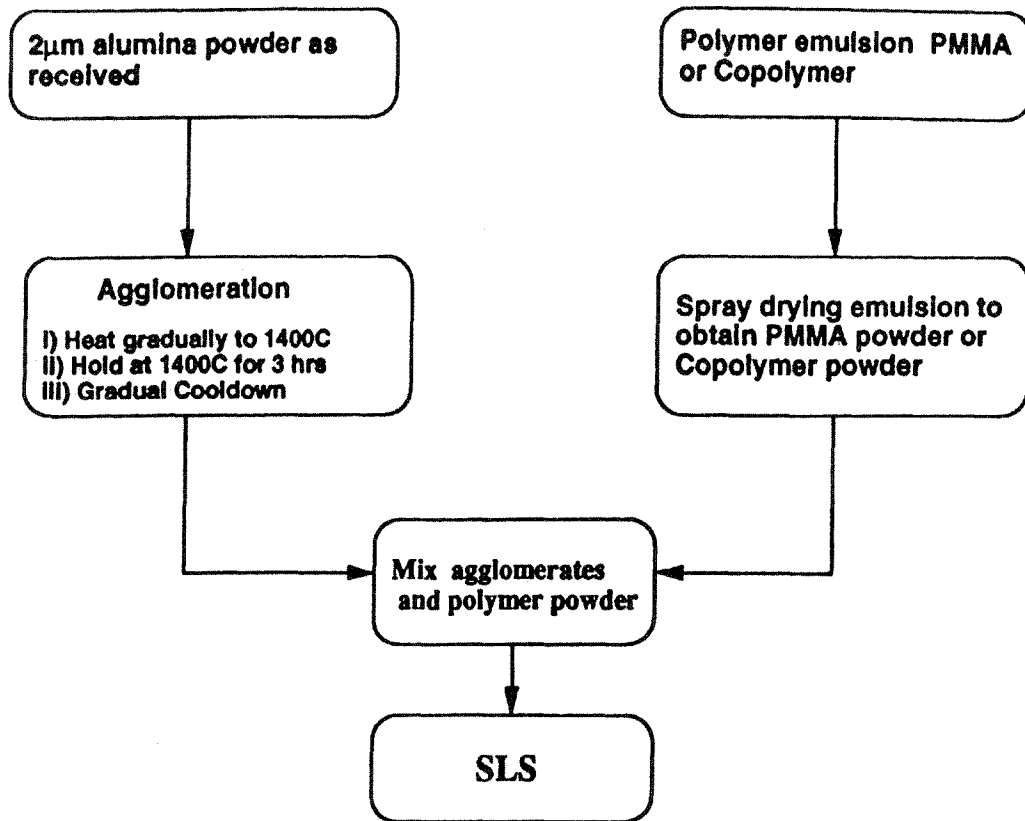


Figure 1. Processing of 2µm Alumina powder

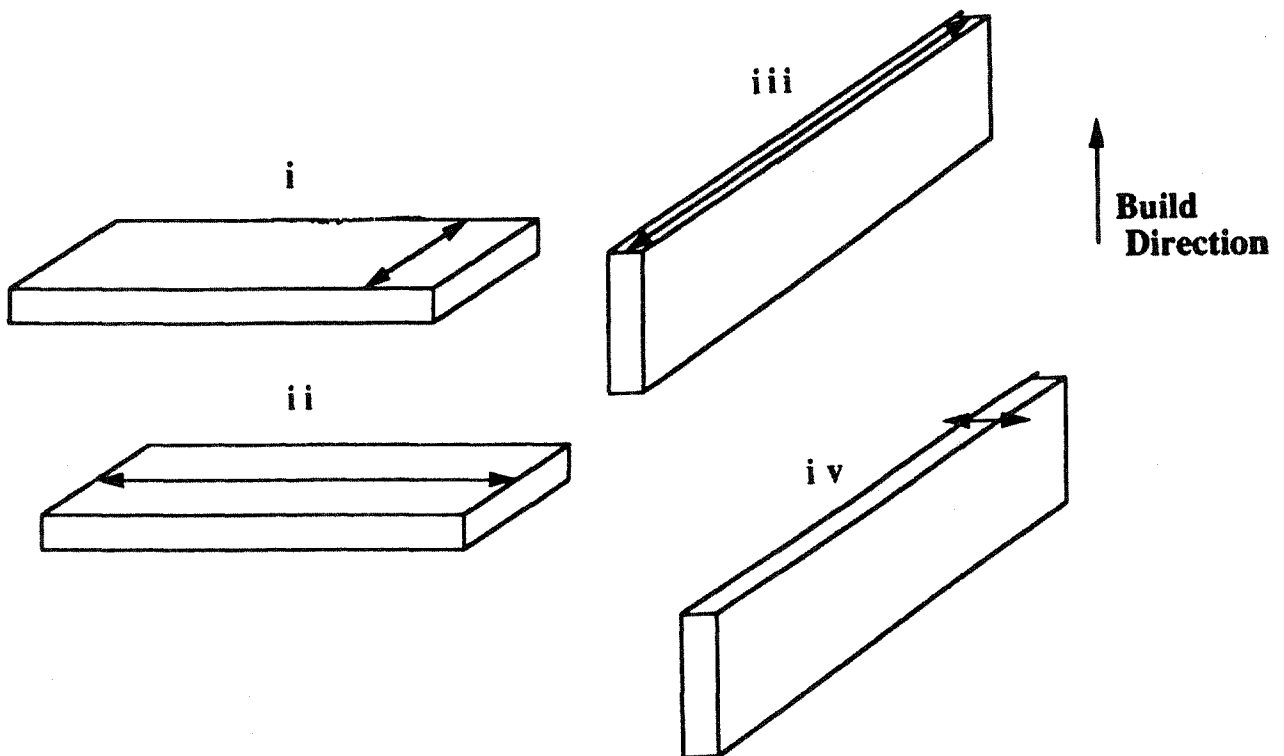


Figure 2. Orientations used in building the bend strength specimens. i: build along thickness, scanning along width ; ii: build along thickness scan along length; iii: build along width with scanning along length; iv: build along width, scan along thickness

The 15 μ m alumina powders were spray dried with the copolymer in a Anhydro spray drier with inlet temperature of 200C and outlet temperature of 80C. The agglomerates of 2 μ m alumina were mixed with the polymer powder obtained by spray drying the polymer emulsion. Two mixtures were made, i) the agglomerates of 2 μ m alumina mixed with the spray-dried PMMA powder and ii) the agglomerates of 2 μ m alumina mixed with the spray-dried copolymer powder. The spray-dried and mixed powders were then SLS processed.

The layer thickness was kept constant at 175 μ m. The laser scanning speed and laser scan line spacing were varied along with the power of the laser to change the incident laser energy density. The scan line spacing was varied between 75 μ m and 125 μ m and the scan speed was varied from 30cm/s to 150 cm/s. The laser power was varied between 6 and 14W. The energy density was varied between 2 cal/cm² and 8 cal/cm². Three point bend strength specimens 0.076m x 0.025m x 0.00625m (3"x1"x0.25") were made by SLS. The specimens were made in 4 orientations i) built along thickness with scanning parallel to width ii) built along thickness with scanning parallel to length, iii) built along width with scanning parallel to thickness and iv) built along width with scanning parallel to length as shown in Fig.2.

Results and Discussion

SLS of parts from both the spray-dried and mixed powders produced strong green parts. For the case of the spray-dried 15 μ m powder built along thickness with scanning parallel to width, condition (i), the variation of density with incident energy density is as shown in Fig.3. The density varies little with incident energy density. The variation of bend strength of the green parts with incident energy density is shown in Fig.4. It may be seen that the green strength increases initially with energy density because of better melting and wetting of the alumina by the molten polymer. At still higher energy densities the strength decreases due to decomposition of the polymer.

Scanning along the length while building along thickness, condition (ii) produces a lower green strength at lower energy densities than those scanned along width and built along thickness, condition (i). This anisotropy is due to the region scanned by the laser having longer time for heat transfer between two consecutive scans for condition (ii). At higher energy densities i.e., around 6cal/cm² the strength of the samples made by scanning along length has a higher strength due to degradation of polymer for condition (i). Even in the case of scanning along length we have a decrease in strength at higher energy densities. But this decrease occurs at still higher energy densities.

With increase in energy density, higher temperatures are reached in the sample being made. As temperature increases well above the glass transition temperature, the degradation of the polymer increases. Temperatures in the decomposition regime are attained at lower energy densities in the case of samples with shorter scan lines because the time between successive scan overlays is smaller. In the case of samples made with longer scan lines the temperatures in the decomposition regime are attained only at a higher energy density because there is some time for the heat to be dissociated between successive scan overlays.

In the case of parts built along width with scanning parallel to thickness, condition (iv), the green strength increases with energy density at low energy densities, Fig.5. At higher energy

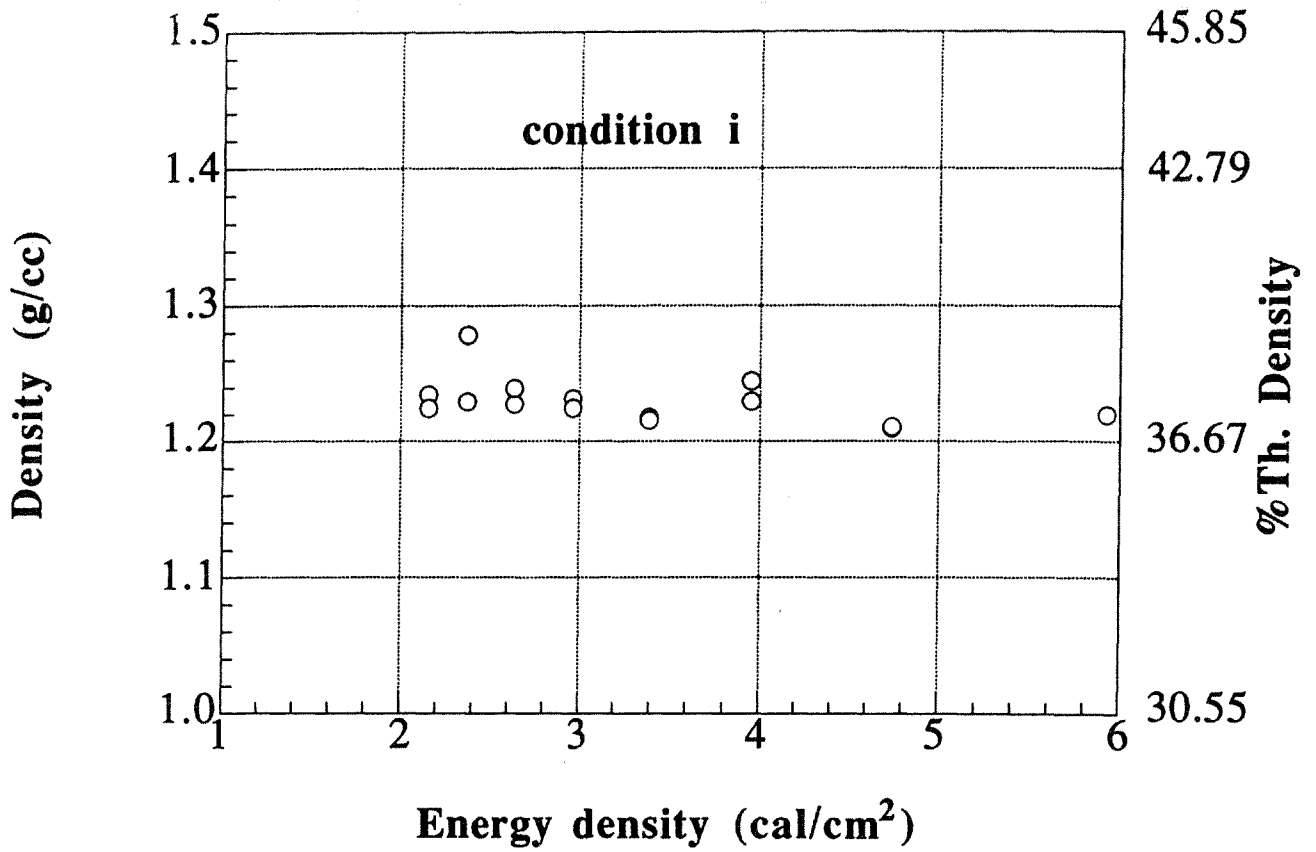


Figure 3. Green density of samples after SLS from 15 μ m alumina coated with copolymer.

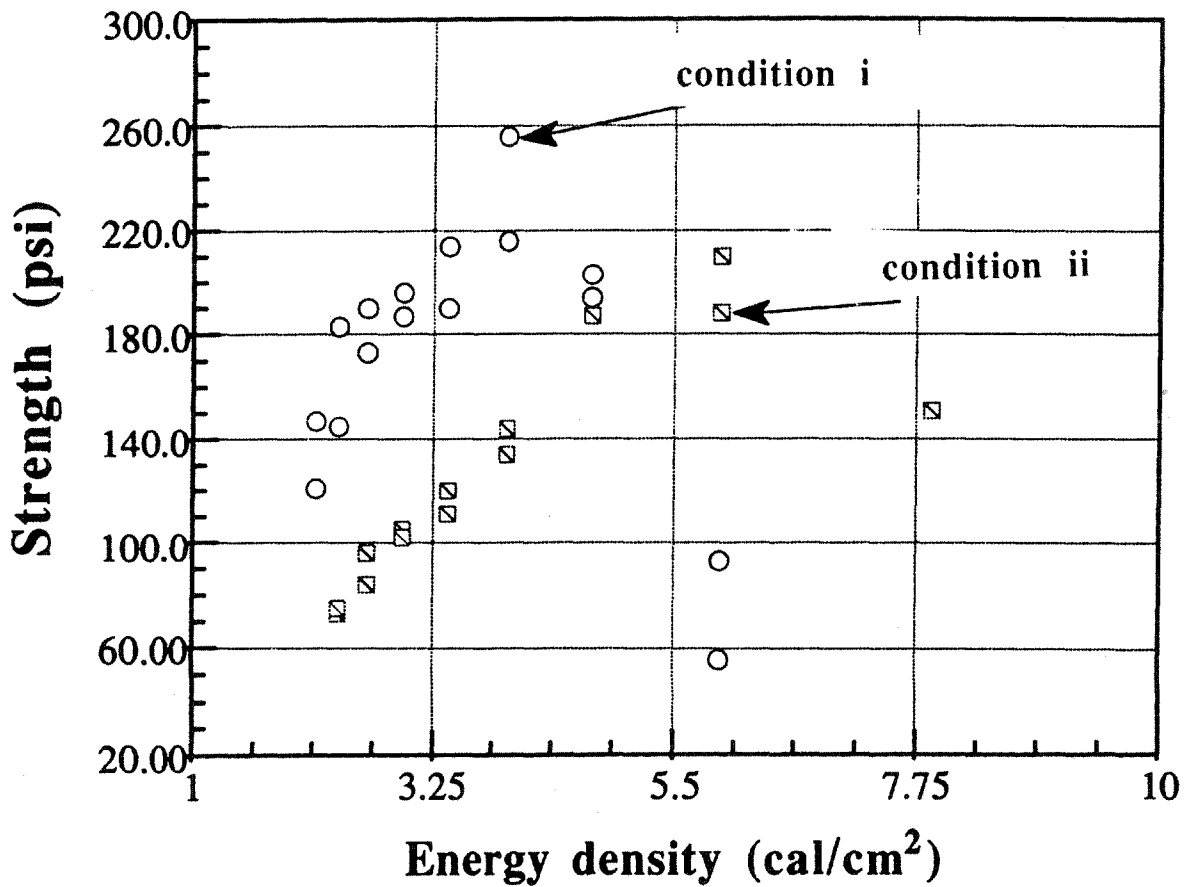


Figure 4. Bend strength of samples from 15 μ m alumina coated with copolymer built along thickness.

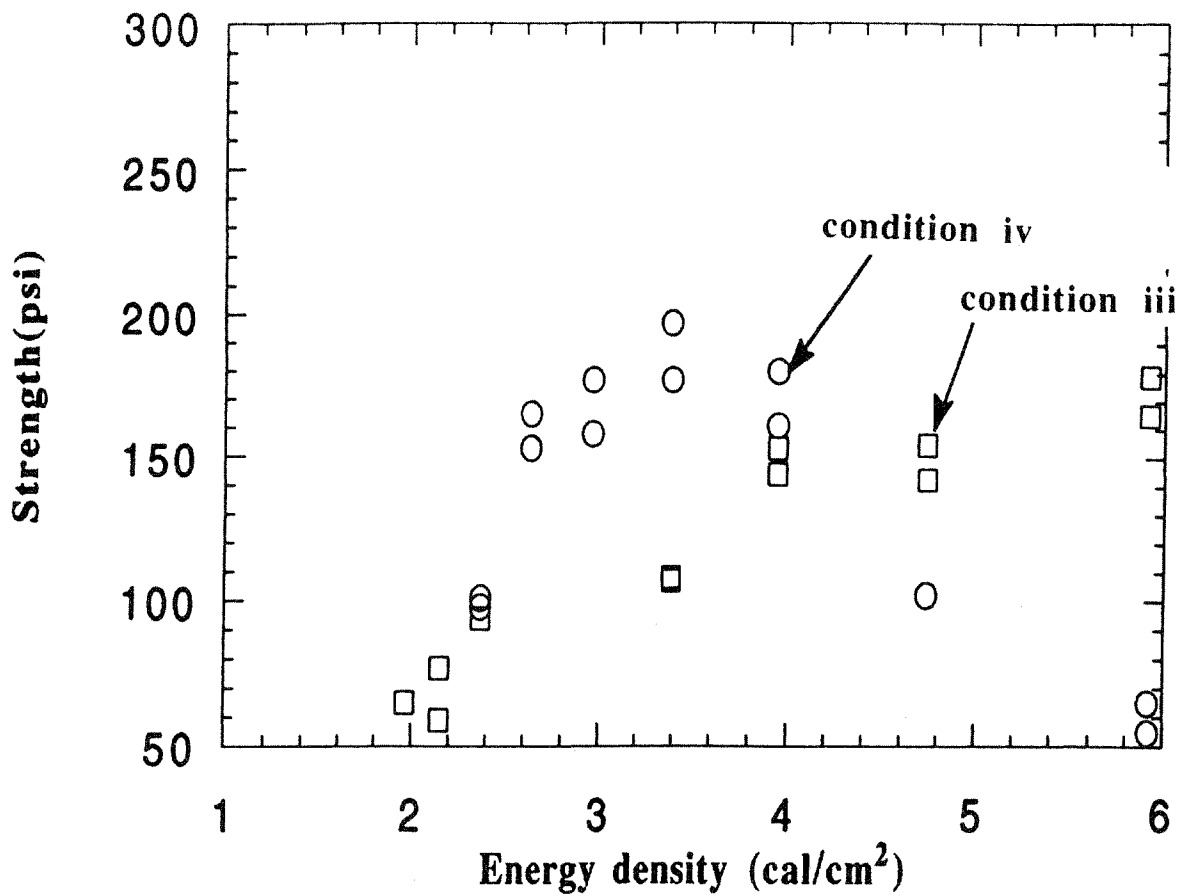


Figure 5. Strength of samples from 15µm alumina coated with copolymer built along width.

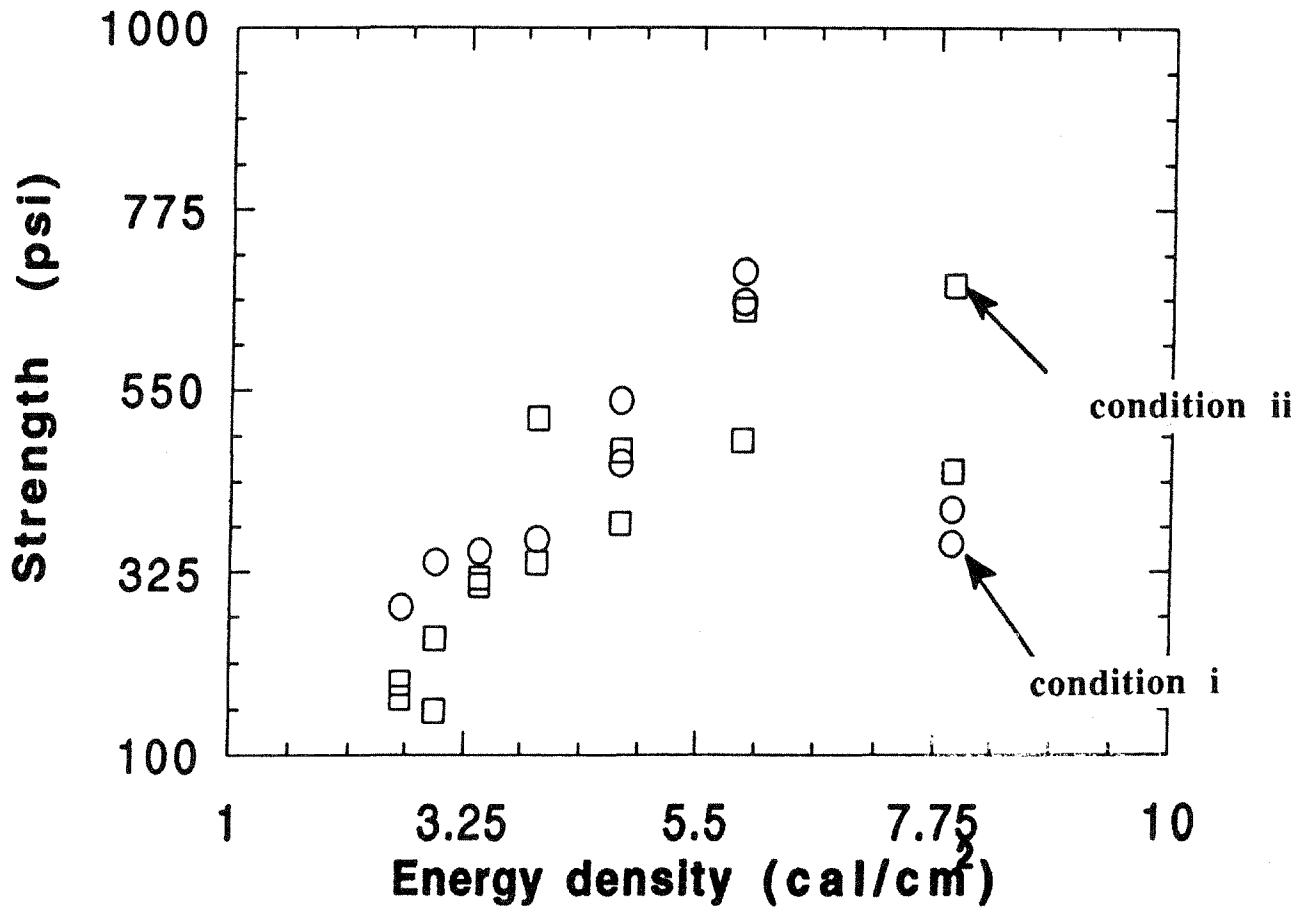


Figure 6. Strength of samples from agglomerated 2µm alumina mixed with PMMA built along thickness.

densities the strength decreases for reasons stated before. Once again it may be seen that when the scan lines are longer the green strength obtained at lower energy densities is lower. Again the peak in the strength vs. energy density plot occurs at a higher energy density for the parts made with longer scanlines.

SLS of agglomerated 2 μ m alumina mixed with 30 V% PMMA.

Specimens for bend-strength were made from this mixture in the four orientations described above. For specimens built along thickness, conditions (i) and (ii), Fig.6 it may be seen that the maximum strength is higher than that obtained for the 15 μ m alumina spray-dried with copolymer. This is due to the fact that the alumina agglomerates are much larger than 15 μ m and hence have a lower surface area to be wet by the polymer. The polymer content is also slightly higher. The variation of green strength with energy density follows similar trend as in the previous case with the strength increasing with energy density at lower energy densities. The peak in the strength vs. energy density plots occurs at a higher value of the energy density for the parts made with longer scan lines.

When specimens were built along the width at low energy densities the parts made with shorter scan lines, condition (iv) had higher green strength, Fig.7. A slight increase in density with increase in energy density may also be observed, Fig.8.

SLS of the mixture of agglomerates of 2 μ m alumina with 20 V% copolymer.

Specimens for bend strength measurement were made from this mixture by building only along the thickness direction. Three different scanning methods were employed. They were i) scanning along width, ii) scanning along length and iii) scanning alternate layers in length and width. The density increases slightly with increase in energy density, Fig.9. Scanning along the length produces samples of lower density compared to the other two modes at lower energy densities. In this case also it may be seen (Fig.10) that the strengths are lower for scanning along the length at low energy densities, as described earlier. The case of mixed (alternate) scanning results in samples of strength intermediate between the two other modes. This is to be expected since alternate layers will have higher and lower strengths corresponding to scanning in the other two modes.

Summary

Successful SLS was done on 2 μ m alumina particles by agglomerating the powders and mixing with polymer powder prior to Laser Sintering. Samples were built in different orientations in the SLS process. In all cases the samples that were built using shorter scan lines had a higher strength at lower energy densities. For both longer and shorter scan lines the green strength increases initially with increase in energy density, attains a peak and then starts decreasing with further increase in energy density due to polymer degradation. The peak in the case of samples built with longer scan lines occurs at a higher value of the energy density due to lower temperatures reached under those conditions.

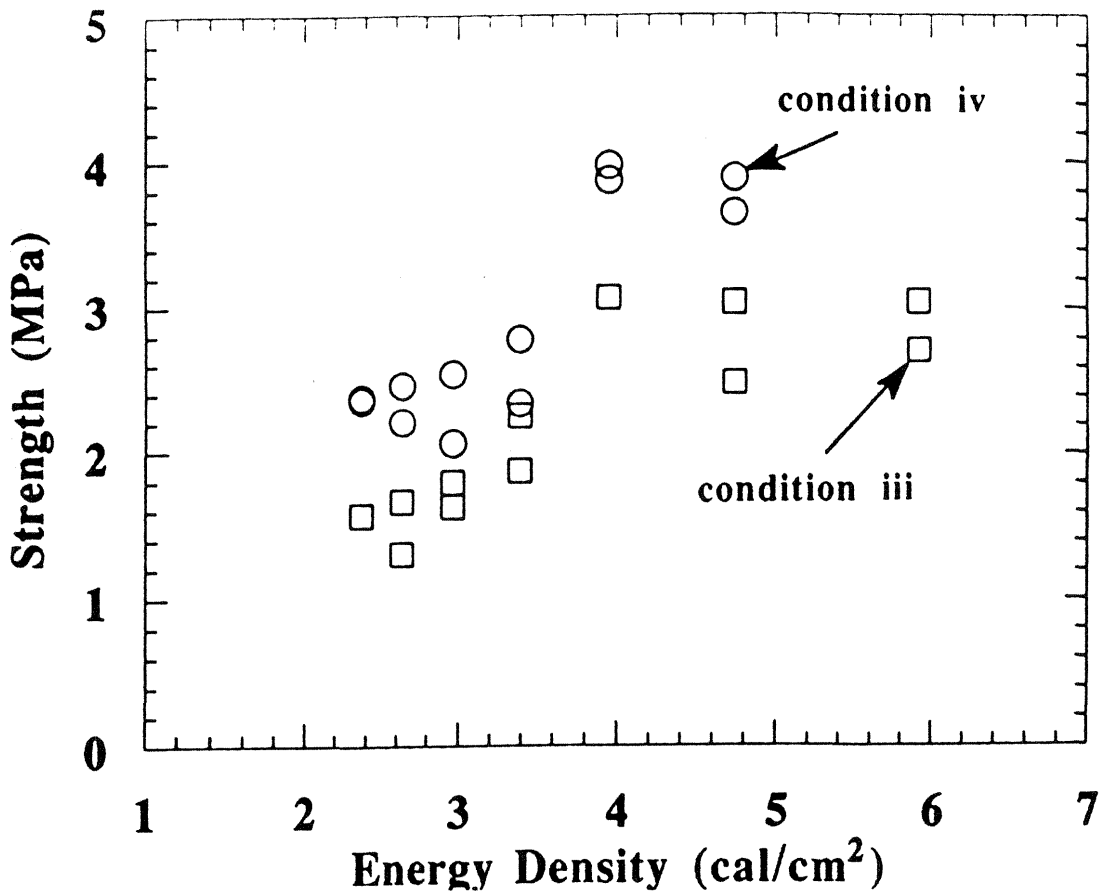


Figure 7. Strength of samples from agglomerated 2 μ m alumina mixed with PMMA built along width.

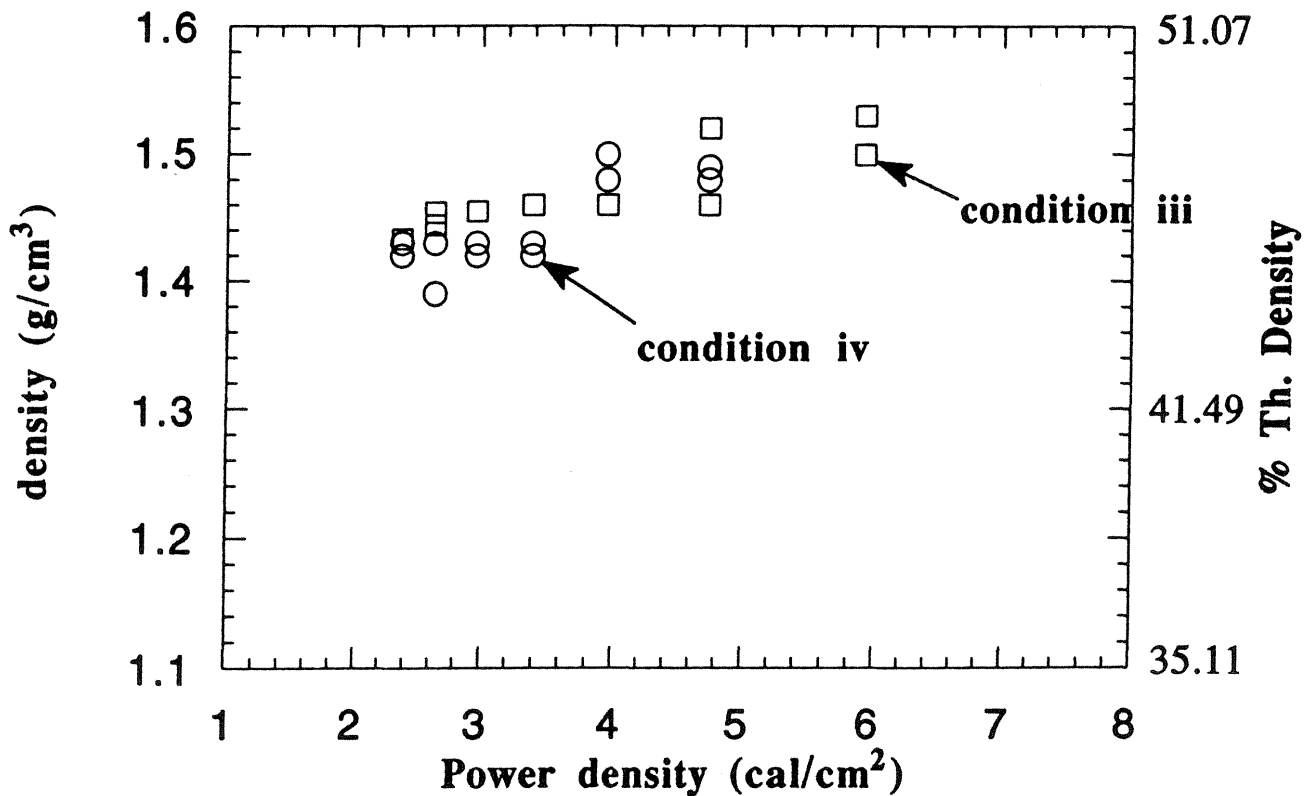


Figure 8. Density of samples as SLS for mixture of 2 μ m alumina mixed with PMMA.

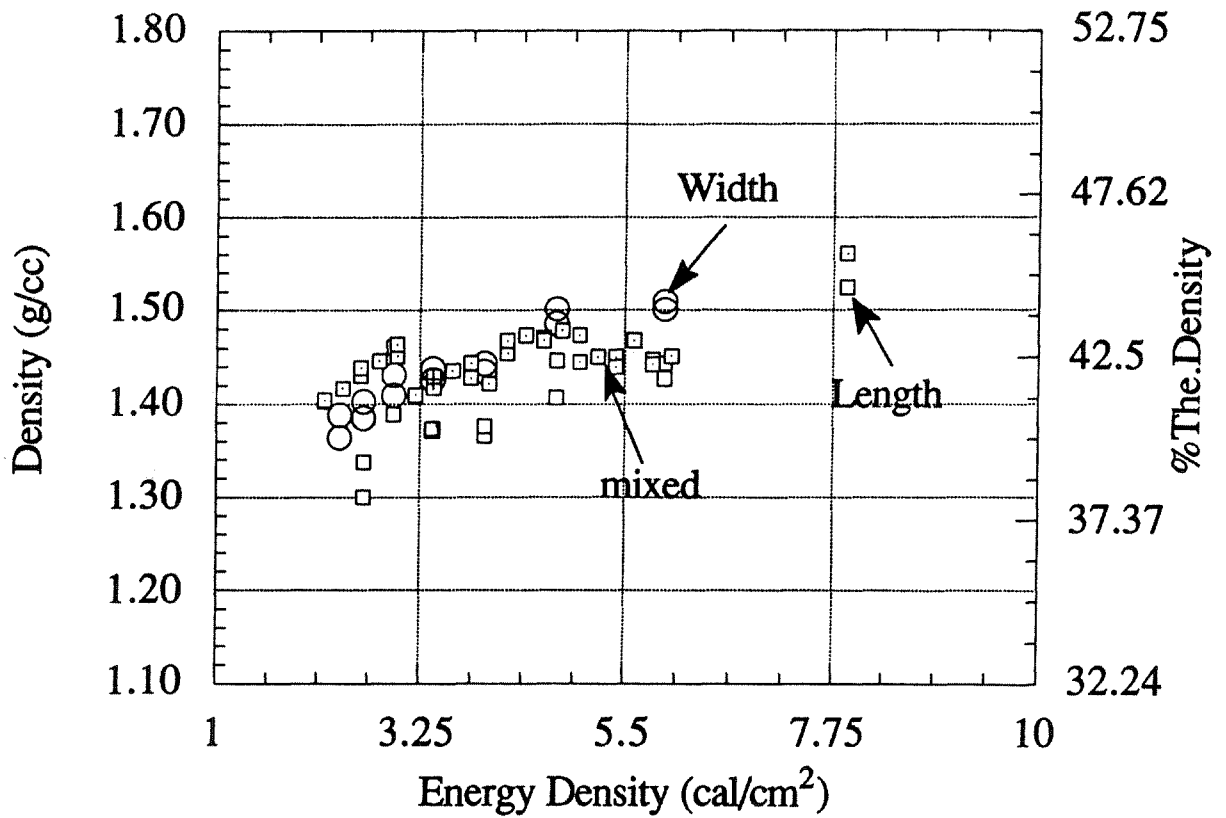


Figure 9. Density of samples from agglomerated 2 μ m alumina mixed with copolymer built along thickness.

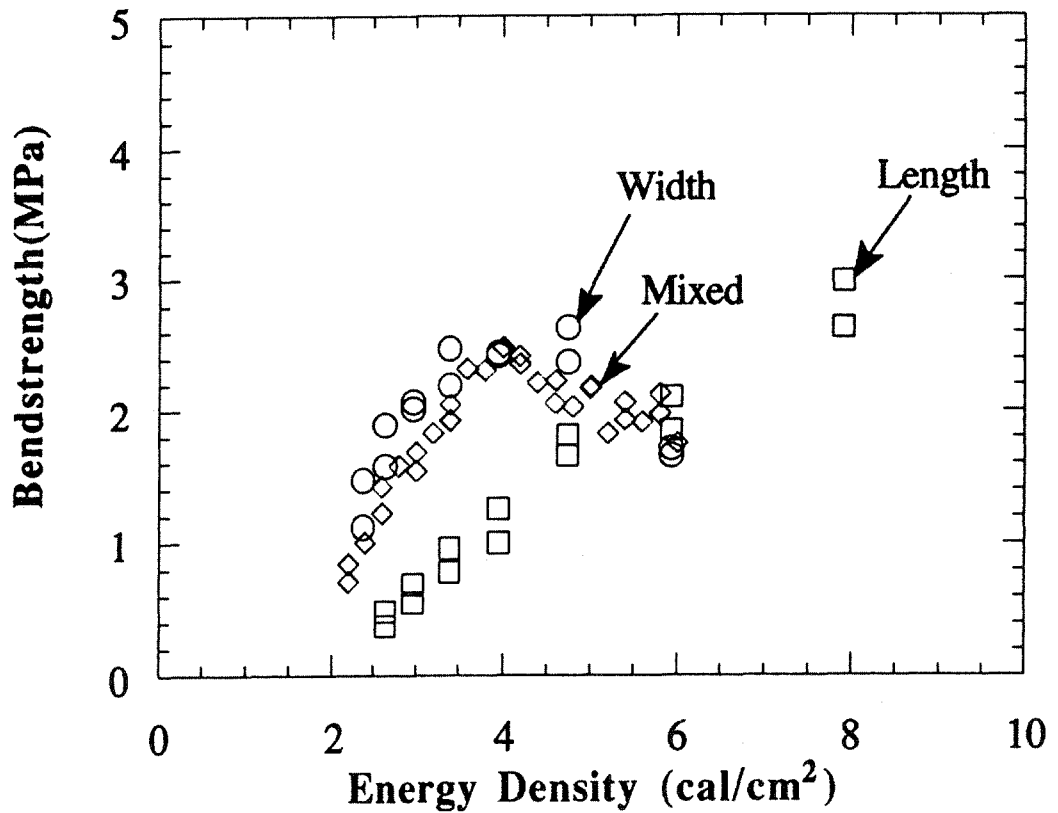


Figure 10. Strength of samples from agglomerated 2 μ m alumina mixed with copolymer built along thickness.

Acknowledgments

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