Full Densed Alumina-Glass Composites by SLS and Ceracon Forging Process

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Selective Laser Sintering (SLS) process has been employed to fabricate alumina-glass composites using monoclinic HBO₂ as an inorganic binder. Subsequent post-thermal processing of green SLS parts at various temperatures yielded glass-ceramic composites. Composites with higher glass contents showed higher density and bend strength for all firing temperatures than those with lower glass contents. For a firing temperature of 900°C, the glass redistribution arising from a better viscous flow of glass gave all composites maximum density and bend strength. In addition, fully dense SLS glass-ceramic parts were obtained by employing the Ceracon forging process. Bend strength of these Ceracon forged composite parts reached as high as 70 to 110 MPa.

1. INTRODUCTION

Solid Freeform Fabrication (SFF) is an advanced manufacturing technology which generates geometrical objects directly from a three-dimensional computer image without part-specific tooling or human intervention [1, 2]. Selective Laser Sintering (SLS) is a form of SFF and employs a focused laser beam which is controlled by a CAD data base to selectively scan a powder bed, binding loose powder, to make a thin layer of bound powder. The desired object is generated by laying down a number of such layers and successively sintering them [3, 4]. The primary advantage of the SLS process is the flexibility of selection of material systems compared to other SFF techniques [5].

There are two kinds of intermediate binders for the SLS of ceramic powders. One is an inorganic binder approach and the other is an organic binder, such as the Poly Methylmethacrylate (PMMA), approach. Ammonium dihydrogen phosphate was employed as an inorganic binder for SLS of alumina [6, 7]. Ammonium dihydrogen phosphate has a low melting point (190°C) and forms a glassy phase around alumina particles under the laser beam. During the subsequent firing step of green SLS part, the ammonium phosphate binder reacts with the alumina, resulting in aluminium orthophosphate (AlPO₄). Aluminum has recently been used as a binder for SLS of alumina [8]. Aluminum melts completely or partially under the laser beam and binds alumina particles. The subsequent heating step of the green part in air oxidizes aluminum into alumina completely. Polymers have been used as an organic binder to fabricate green SLS ceramic parts [9, 10]. The dimensional accuracy of a ceramic/polymer composite bound with polymer is excellent. However, the polymers have to be removed thoroughly at low temperature before further sintering at high temperature. This process is associated with long and careful debinding steps.

Monoclinic HBO₂ has recently been developed for an inorganic binding approach for the SLS of alumina [11]. Due to its low viscosity and better wetting of the alumina particle surface, there is better feature definition of green SLS parts and higher bending strength of green and fired parts, compared to those made with other inorganic binders such as aluminum and ammonium phosphate.

Since alumina reacts well with zinc borosilicate glass that has a low softening point (630°C), fabrication of alumina-glass composite parts by the SLS process seems attractive. However, it was found experimentally that the direct use of zinc borosilicate glass as an inorganic binder for the SLS of alumina in a conventional low temperature system led to curling of the newly sintered layer, and hence a debonding between the layers. The alternative approach is to employ monoclinic HBO₂ as an inorganic binder for the SLS of a binary alumina-glass
composite system.

The selection of monoclinic HBO$_2$ as an inorganic binder for the SLS of an alumina-zinc borosilicate glass powder blend offers the possibility of obtaining a desired shape with alumina-glass composites. Crystalline monoclinic HBO$_2$ can transform completely into amorphous boron oxide during the laser scanning. Densification and strengthening of the green SLS composites can occur through both the viscous flow of boron oxide and glass, as well as the reactions among alumina, glass, and boron oxide during post-thermal processing. Chemical reactions during heat treatment can lead to the formation of new crystalline phases. Thus, fabrication of glass-ceramic composites is possible by SLS and post-processing of alumina-glass-monoclinic HBO$_2$ powder blends.

This paper deals with the fabrication of glass-ceramic composites through the post-processing of green SLS parts that were made with alumina-zinc borosilicate glass composite and monoclinic HBO$_2$ as an intermediate binder. Two kinds of post-processing are employed; (i) simple heat treatment, and (ii) the Ceracon forging process. The Ceracon process [12] is a patented, quasi-isostatic, hot consolidation technique that employs a ceramic particulate material as a pressure transmitting medium instead of a gas media used in Hipping (Hot Isostatic Pressing). The Ceracon process is a low-cost powder metallurgy process for achieving near-net-shape, full density parts without the requirement of containing the specimen. Fig. 1 shows the schematic diagram of this process. The maximum pressure which can be applied is 200 Ksi. The process covers a broad range of metallic, ceramic, and polymeric materials and composites. Direct application of the Ceracon forging process to a preform, fabricated by the SLS process, is expected to reduce the time involved in the manufacturing of both prototype and net-shape powder metallurgy parts. The effects of materials and processing parameters on the physical and mechanical properties of final glass-ceramic parts are described in this paper.

2. MATERIALS AND EXPERIMENTAL PROCEDURE

High purity, electronic grade, 15 μm diameter, aluminum oxide powder provided by Norton Materials Corporation and a 60 mesh (250 μm) 99% boron oxide powder from Johnson Mathey were the starting materials. The initial boron oxide powder of 250 μm diameter was ground by a Szegvari attritor system and sieved to less than 75 μm.

Zinc borosilicate glass powder (400 mesh) was provided by Transene Company. The range in chemical composition of the glass is shown in Table 1. The softening point of this glass is around 630°C and the true density is between 2 and 3 g/cm³. Most of the particle size is less than 10 μm and particle shape is irregular as revealed by SEM micrograph.

Zinc borosilicate glass powder was isothermally heat treated at 600°C, 700°C, 800°C, and 900°C for 6 hours to investigate the crystallization behavior. In addition, Differential Thermal Analysis (DTA) was carried out on zinc borosilicate glass powder to determine its crystallization temperatures. In this experiment, the alumina crucible containing the glass powder was heated from room temperature up to 1000°C with a heating rate of 10°C/min and pure alumina was used as a reference material.

Alumina and glass powders were mixed in the ratio of 1:1, 7:3, and 9:1 by weight. Each powder mixture was blended with B$_2$O$_3$ in the ratio of 3:1 by weight. The pre-baking-out procedure to transform B$_2$O$_3$ to Monoclinic HBO$_2$ was described in the SLS of an alumina-boron oxide system [11]. Baked-out powder blends were immediately sintered in a SLS system of the University of Texas at Austin. Test specimens with dimension of 0.076

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration (wt.%)</th>
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<tbody>
<tr>
<td>ZnO</td>
<td>&lt; 60%</td>
</tr>
<tr>
<td>B$_2$O$_3$</td>
<td>&lt; 40%</td>
</tr>
<tr>
<td>Amorphous SiO$_2$</td>
<td>&lt; 20%</td>
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