Effect of Bimodal Powder Distribution on Mechanical properties and Biological Activity of Hydroxyapatite and its Composites with Silicon Nitride Processed by Ceramic Fused Filament Fabrication (CF^3)

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Abstract
In this work we have prepared hydroxyapatite (HAp) and its composite parts with 0, 5 and 10 wt.% Si_3N_4 using the ceramic fused filament fabrication (CF^3) process that uses filaments containing HAp ceramic powders and polymer binder to print green parts followed by debinding and sintering. Feedstocks with 40 vol.% ceramic powder were prepared followed by filament extrusion for printing. A three-step thermal debinding cycle developed using thermogravimetric analysis of the feedstock resulted in complete removal of the binder and a sintered density of ~85% was achieved in all samples. Almost 70% reduction in the grain size was observed with addition of Si_3N_4 to HAp due to the formation of liquid phase during sintering. In-vitro testing was performed on the sintered samples of pure HAp and HAp with Si_3N_4 using MC3T3-E1 cells to identify cell proliferation, differentiation and mineralization caused due to addition of Si_3N_4. We observed that after three weeks of osteogenic induction, a minimum of 10wt.% Si3N4 is needed to have significant biological performance compared to pure HAp.

Keywords: Fused filament fabrication; Material extrusion; Hydroxyapatite; Silicon nitride; Ceramic printing; Osteoblast; Biological testing;

1. Introduction

Processing of ceramics using conventional manufacturing techniques typically involve powder pressing followed by high-temperature sintering and machining, as desired. However, machining of ceramics to complex shapes is not only difficult, due to their inherent brittleness, but also causes significant amount of tool wear and material waste. Therefore, advanced manufacturing technologies such as additive manufacturing (AM) have been widely being adopted to manufacture wide variety of ceramic components [1–4]. The AM technologies that are being used to process ceramics can be grouped as slurry-based (stereolithography, inkjet printing, direct ink writing, digital light processing), powder-bed based (selective laser sintering/melting, binder jetting) and material extrusion-based (fused filament fabrication, robocasting). Although each technology provide unique processing flexibility and capabilities to manuscript complex ceramic parts, ceramic fused filament fabrication (CF^3) is most economical as the machine/equipment, feedstock materials and overall processing cost is relatively lower than other AM processes [3,4]. Further, the elimination of loose powder and liquid in CF^3 process makes it a best process