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Selective laser sintering of hydroxyapatite reinforced polyethylene composites for bioactive implants and tissue scaffold development

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Abstract: Selective laser sintering (SLS) has been investigated for the production of bioactive implants and tissue scaffolds using composites of high-density polyethylene (HDPE) reinforced with hydroxyapatite (HA) with the aim of achieving the rapid manufacturing of customized implants. Single-layer and multilayer block specimens made of HA–HDPE composites with 30 and 40 vol % HA were sintered successfully using a CO₂ laser sintering system. Laser power and scanning speed had a significant effect on the sintering behaviour. The degree of particle fusion and porosity were influenced by the laser processing parameters, hence control can be attained by varying these parameters. Moreover, the SLS processing allowed exposure of HA particles on the surface of the composites and thereby should provide bioactive products. Pores existed in the SLS-fabricated composite parts and at certain processing parameters a significant fraction of the pores were within the optimal sizes for tissue regeneration. The results indicate that the SLS technique has the potential not only to fabricate HA–HDPE composite products but also to produce appropriate features for their application as bioactive implants and tissue scaffolds.

Keywords: selective laser sintering, hydroxyapatite, polyethylene, bioactive implants, tissue scaffolds

1 INTRODUCTION

Hydroxyapatite (HA) is a ceramic to which bone bonds [1]. When combined at high filler levels with a binding polymer, the material forms a bioactive composite. HA particulate reinforced high-density polyethylene (HDPE) composites (HA–HDPE) were developed by Bonfield *et al.* [2, 3] and have been used clinically for bone augmentation for over 15 years [4]. The clinically used grade of HA–HDPE composite, known commercially as HApEXTM, is an analogue material designed to replace bone. This material has been used for orbital floor implants for patients suffering from post-enucleation socket syndrome and in middle ear implants [5]. Presently, these implants are shaped by conventional tech-

niques such as moulding and machining. These indirect techniques require more process steps, are less automated, have high material waste in the case of machining, and require individual tooling for one-off jobs in the case of moulding. These conventional techniques also impose geometrical restrictions on the shapes that may be produced, i.e. undercut and re-entrant shapes are difficult to produce from moulds, the shape of articles produced by cutting techniques are restricted by the movements required by the cutting tool, and neither technique can produce internal structures. The limitations of conventional manufacturing methods therefore restrict the production and application of HA–HDPE.

Selective laser sintering (SLS) is an additive manufacturing process, which belongs to a family of techniques known as layer manufacturing (LM). The use of layer manufacturing allows physical parts to be created immediately, directly, and automatically from a three-dimensional representation known as a three-dimensional computer-aided-design (CAD)

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model, like the information obtained from a computed tomography (CT) or magnetic resonance imaging (MRI) scan. Layer manufacturing was used for rapid prototyping (RP) and has emerged as a key enabling technology for rapid manufacturing (RM) to produce directly usable products or parts. RP produces parts for prototyping or tooling purposes, while RM pursues the production of objects for long-term use. RM facilitates total customization of products and one individual product to be manufactured as easily as millions of products (i.e. mass production). Therefore, the layer-based RM approach permits extremely complex shapes to be produced and enables the economically viable production of implants that are customized with respect to size and functionality for individual patients. Customized implants give the potential to improve bone defect repair methods and enhance the longevity of the implants by providing a more secure fit due to exact geometry [6]. For SLS fabrication of polymer/ceramic composite powders, the polymer serves as a binder which is activated at relatively low temperature, while the ceramic serves as a structural and/or a functional material. During the SLS process, powders are subjected to low compaction forces, and thus fabricated objects are inherently porous, a key property requirement for some *in vivo* implantation devices. The nature and extent of this interconnected porosity may be tailored and controlled to meet application criteria through material selection, physical design, and processing parameters. Furthermore, SLS has the potential capability to produce functionally graded materials (FGMs) that exhibit spatially inhomogeneous properties and microstructures, allowing for the positional variation of physical properties and characteristics. This functional grading would allow new products of unique and customized characteristics to be produced. Functional grading is possible in SLS by varying the composition of two or more powdered materials across the surface, interface, or bulk of the material during the build sequences. Possible applications include encouraging locational bone ingrowth by greater density of HA and providing different mechanical properties in the areas of high load bearing or wear by the use of different polymers.

To date, medical applications of SLS have primarily concerned the production of physical models of human anatomy for surgical planning, training, and design of customized implants. Berry *et al.* [7] built SLS models of two skulls and a normal femur using nylon, with the dimensions being in good agreement with the CT data. However, commercially available SLS materials are not biocompatible and cannot be

used for *in vivo* applications. Rimell and Marquis [8] demonstrated that SLS has the potential to fabricate linear continuous ultrahigh molecular weight polyethylene (UHMWPE) solid bodies for clinical application by using a non-commercial SLS machine without preheating the powders. During the laser processing, material shrinkage caused problems in forming sheet-like structures, and degradation of UHMWPE was unavoidable. Scaffolds with periodic cellular and biomimetic architectures were designed and fabricated by SLS by Das *et al.* [9] using nylon-6. Williams *et al.* [10] fabricated porous scaffolds of polycaprolactone (PCL), a bioresorbable polymer, with the potential application for bone and cartilage repair by SLS. The early application of SLS for composite implant fabrication using HA and other calcium phosphate materials [11, 12] involved coating of the HA powder with a secondary polymeric binder, poly(methyl methacrylate) (PMMA). Problems were encountered in the use of organic solvents (diluted methanol) in coating and additional post-debinding and sintering. Tan *et al.* [13] and Chua *et al.* [14] have investigated scaffolds produced with physically blended HA–polyether ether ketone (PEEK) and HA–polyvinyl alcohol (PVA) composites using a commercial SLS system.

The present work investigated the potential of SLS to fabricate parts using 30 and 40 vol % HA–HDPE composites and analysed the effects of laser power and scanning speed on the sintered parts, the features of pores, and the HA particle morphology.

2 METHODOLOGY

2.1 HDPE and HA raw materials

The HDPE (Rigidex HM4560XP) was supplied in pellet form by BP Chemicals Limited. Synthetic HA particles (P218R, Plasma Biotol Limited, Tideswell, UK) were used as the filler material. The particles had a median size, $d_{0.5}$, of 3.80 μm and a specific surface area of 13.54 $\text{m}^2 \text{g}^{-1}$, while the theoretical density was 3160 kg/m^3 [15]. The melting temperature of the HA–HDPE composites was determined as 136 °C by thermal analysis using differential scanning calorimetry (DSC) (DSC 60, Shimadzu, Japan).

2.2 Production of HA and HDPE composite powders

As described in detail by Wang *et al.* [16], the HA and HDPE powders were blended to produce mixtures with 30 and 40 vol % HA. These mixtures were compounded in a twin-screw extruder (Betol BTS40L,

Betol, Luton, UK) to produce the HA–HDPE composites. The extruded composites were subsequently pelletized in a Betol pelletizer. The pelletized HA–HDPE composites were powderized in a Retsch ultra-centrifugal mill (Retsch, Hann, Germany) using a sieve of 0.250 mm aperture size. Liquid nitrogen was used as a coolant to prevent temperature increases and to embrittle the material during milling. It should be noted that the compounded composites prepared in this study are matrix composites containing both the binder and structural material, not a distinct component mixture in which the binder is separate from the structural material. The sintering of matrix composites can avoid the segregation of the different elements that can occur with mixed-element blends.

2.3 SLS systems and processing

Instead of using a commercial SLS system, an experimental SLS system was developed by incorporating an 'experimental powder bed chamber' and a CO₂ laser system (Synrad Limited, Washington) that has an average output power of between 0.2 and 10 W and can be driven at various scanning speeds of between 0.2 and 10 000 mm/s. This system permits the economical use of the expensive material and greater variation of laser sintering parameters than commercial systems. A laser beam with a wavelength of 10.6 μm was focused to a spot size of 193 μm at a focal length of 491 mm and used to scan the powders. As shown in Fig. 1, the experimental powder bed chamber simulates the commercial SLS processing chamber but has a smaller powder bed volume (103 mm diameter cylinder for both operation and feed powder bed). During sintering, nitrogen gas filled the chamber to create an inert

Fig. 1 Photograph of the experimental chamber showing the build and feed cylinders and feed mechanism

atmosphere and the oxygen content was maintained below 5 per cent. One heater band enclosed both the feed and build cylinders and one overhead heater installed at the top of the chamber preheated the powders to 110 °C. The dimensions of the sintered specimens were 4 × 23 mm, and the laser scan space was 63 μm . To begin a detailed investigation, single-layer specimens were built over a wide range of laser parameters to analyse the effects of laser power and scanning speed on the sintered HA–HDPE composite. Then, optimum parameters were used to build specimen blocks of five layers with a layer thickness of 0.4 mm.

2.4 Characterisation

The height of sintered single-layer specimens was measured by Vernier calipers. The mean value of three measurements was recorded. The particles of HA–HDPE composite powders, the surface morphologies of the sintered single layers, and the cross-section of scalpel-cut sintered blocks were examined using a scanning electron microscope (SEM) (LEO 440, Cambridge, UK). All samples were sputter coated with gold prior to imaging.

3 RESULTS AND DISCUSSION

3.1 Microscopic examination of HA–HDPE composite powders (non-sintered)

Figure 2 shows the HA–HDPE composite particles of 30 and 40 vol % HA. It can be seen that most of its particles were of irregular shape and demonstrated a wide range of particle sizes. Some elongated filaments are also present among the particles. There are no substantial differences in shape between the composite particles of 30 vol % HA and 40 vol % HA.

3.2 Effect of SLS parameters on the sintering of HA–HDPE composites

3.2.1 Visual effects of laser scanning speed and laser power

The quality of SLS fabricated parts is influenced by many factors such as materials properties, powder bed characteristics, and process parameters. Of the process parameters, laser power and scanning speed were found to be the two most influential parameters. Williams and Dechard [17] studied the sintering height/depth of powders following single-pass laser scanning and showed that there is a minimum power necessary to induce sintering and a maximum scanning speed above which sintering does not

Fig. 2 SEM micrograph of (a) 30 vol % HA content, (b) 40 vol % HA content composite powders

occur. The sintering height to which the laser is able to sinter determines the maximum layer thickness that can be used to construct a part. In order to investigate the effect of the laser scanning speed, the speed was varied from 300 to 4800 mm/s, while the laser power was fixed at 3.6 and 7.2 W. As shown in Fig. 3, the scanning speed had a significant effect on the height of a single sintered layer and decreased with increasing laser scanning speed. This result may be because, at lower scanning speed, a relatively longer interaction time leads to greater energy absorption and more time for particle interaction and fusion. The surface of a single layer sintered at a scanning speed of 300 and 600 mm/s changed colour from white (the colour of HA–HDPE powders) to dark-brown, indicating that the HDPE on the composite surface had degraded. At low scanning speeds, the composite powders experienced a longer interaction time which would result in the degradation of the composite powders even in the low-oxygen environment. For the high laser scanning speed of 4800 mm/s, no single layer could be sintered under 3.6 W, and a single layer sintered under 7.2 W was

fragile. Owing to the short interaction time at the high scanning speed, the particles did not have enough time to interact and fuse. Therefore, these initial tests demonstrated that it was better to use a relatively moderate scanning speed to sinter HA–HDPE composites.

A laser scanning speed of 1200 mm/s was chosen to investigate the effect of laser power over a wider power range, owing to the success of layers being sintered at this speed at both 3.6 and 7.2 W. As shown in Fig. 4, the height of the single layer increased with increasing laser power. At the same scanning speed, a higher laser power led to more energy absorbed in the composite powders and thus a higher degree of sintering. At a laser power of 1.2 W, the particles were not well sintered and a continuous single layer was not produced. At a laser power of 2.4 W, a continuous single layer was produced, but the resulting specimen was fragile. When the laser power was above 3.6 W, the single sintered layers were strong enough to be handled. At laser powers between 3.6 and 6.0 W, the colour of the single layer remained similar to that of original composite powders. However, when

Fig. 3 Effect of laser scanning speed on the height of the sintered single layers at 3.6 and 7.2 W power density (30 vol % HA composite powder)

Fig. 4 Effect of laser power on the height of the sintered single layers (30 vol % HA composite powder)

the laser power was increased to 7.2 W, the colour became brown, which was more pronounced with increasing laser power, indicating that the powders are likely to have degraded during processing at the higher laser power. A brown colour has also been observed in previous work on SLS-sintered PVA with different processing parameters [14].

3.2.2 Surface morphology of single sintered layers

Figure 5 shows the surfaces of single sintered specimens built with different power densities at a scanning speed of 1200 mm/s. The typical surface

morphologies of sintered single layers are described in Table 1. Generally, the fusion of particles increased with increasing power. The greater the energy imparted to the powder, the greater is the particle fusion. Individual particles and a few incidences of contact necking were observed at a power of less than 4.8 W. Conversely, complete fusion made the boundaries of particles indistinguishable at a power above 7.2 W. Pores occurred in the layers, and the amount of pores decreased as the degree of particle fusion increased. At a power of 2.4 W, a number of pores were observed measuring 200–400 μm across.

Fig. 5 SEM micrograph of the surface of a sintered single layer with the 30 vol % HA composite and a laser power of (a) 2.4 W, (b) 3.6 W, (c) 4.8 W, (d) 6.0 W, (e) 7.2 W, and (f) 9.6 W at the scanning speed of 1200 mm/s

Table 1 Descriptions of the surface morphology of single sintered HA–HDPE layers under different laser powers at a scanning speed of 1200 mm/s

Laser power (W)	Figures	Surface morphology of single sintered layers
1.2		No layer was sintered
2.4	Fig. 5(a)	Individual particles could be identified; particles fused partially; a few incidences of contact necking were observed between the particles; a large number of pores were seen between the particles; the size of these pores was irregular and varied from small to as large as 200–400 μm
3.6	Fig. 5(b)	Some individual particles could be identified; most of the particles fused with other particles and partial boundaries between the particles were indistinguishable; a large number of pores existed
4.8	Fig. 5(c)	Some individual particles could be identified; most of the particles fused and internecked with each other; large sections of the boundaries between the particles were fused; pores presented between the particles, with internecking
6.0	Fig. 5(d)	Few individual particles could be identified; most of the particles fused together; a few pores were observed
7.2	Fig. 5(e)	Individual particles could not be identified; all particles fused together; boundaries between most particles became indistinguishable; large voids were observed
8.4		Similar to that observed for a power of 7.2 W
9.6	Fig. 5(f)	Individual particles could not be identified; all particles fused together; boundaries between particles became indistinguishable

When the laser power was increased to 6.0 W, the particle boundaries fused together and the pore size decreased considerably, suggesting that the particles would have experienced melting and the particles would have coalesced during the laser sintering. However, large voids, which may have been caused by shrinkage accompanied with the fusion of neighbouring particles, were observed in some areas of the layers sintered at high powers, such as 7.2 W, as shown in Fig. 5(e). Some large HA particles were exposed on the surface of layers sintered at high power, such as 9.6 W, revealing that the HDPE would have been vaporized partially during the processing, leaving HA particles on the surface. It was found by Rimell and Marquis [8] that the laser sintering of UHMWPE at high laser power resulted in volatilization due to a major physical breakdown of the polymer chains to a lower-weight material, some of which vaporizes.

That the size and number of pores in the composite layer varied with laser power and that generally, the porosity decreased with increasing laser power implies that the porosity of HA–HDPE composite parts could be controlled by the energy imparted to the powder surface. A number of other parameters, such as scanning speed, scan space, scan pattern, beam spot size, and layer thickness, may also be methods of controlling this feature and the extent of the pores in HA–HDPE, as these parameters would also influence the energy imparted to the powder surface. Such SLS processing parameters were described by Leong *et al.* [18] as a potential means to fabricate porous polymeric matrix drug

devices. Determining the effect of the other parameters is the subject of further work.

3.3 Morphology of HA particles in the composite following laser sintering

For HA-reinforced HDPE composites, the HDPE provides a binding matrix and the HA provides the bioactivity of the composites. Hence, it is essential to expose HA materials on the surface of the sintered products to allow the biological tissue to interact with them when the composite products are used as bioactive implants or tissue scaffolds. Figure 6 shows the morphology of HA particles on the surface of sintered composite particles. At a laser power of 2.4 W, many small particles were observed on the surface of the sintered products, but most of them seemed to be entrapped or embedded in the sintered surface [Fig. 6(a)]. With the laser power increased to 3.6 W, some HA particles appeared on the surface [Fig. 6(b)]. A large HA particle with a size of approximately 20 μm was exposed. It is believed that laser sintering resulted in the partial surface melting of the composite matrix with HA particles exposed at the surface. At 4.8 W, many HA particles were observed on the surface [Fig. 6(c)], and the surface of the composite melted completely. At 9.6 W, large amounts of small HA particles and numerous larger HA particles are visible [Fig. 6 (d)], indicating that the HDPE may have degraded and vaporized, exposing the HA particles. Therefore, it has been shown that SLS can expose HA particles on the sintered products. In the SLS process, the high power

Fig. 6 SEM micrograph of the morphology of HA on the surface of sintered particles with the 30 vol % HA composite and a laser power of (a) 2.4 W, (b) 3.6 W, (c) 4.8 W, and (d) 9.6 W at the scanning speed of 1200 mm/s

intensity focus provided by a small laser beam spot and overlapping of the beam tracks on the surface could result in local effects on the substrate material, depending on the laser power intensity. At a relatively low power, the effect induced by the laser may occur mainly on the surface of the particles or cause partial melting of the matrices. With increased laser power, the local heat induced by the laser could cause complete melting and generate an effect on the whole of the composite particles. When the laser power is too high, it could result in the degradation or vaporization of the polymer (HDPE). A previous study by Rimell and Marquis [8] showed that ultrahigh molecular weight polyethylene underwent degradation following the SLS process. However, the nature of degradation of UHMWPE is comparable with that produced by irradiation sterilization and may not degrade the properties to an unacceptable level for some applications. In addition, the air trapped inside the particles and between the particles would be released during the evaporation and would undergo a high-temperature exothermic reaction resulting in local burn sites, which could result in the vaporization of the HDPE and expose the HA particles on the surface.

As Kasemo and Lausmaa [19], among others, have described, biological tissues interact mainly with the outermost atomic layers of an implant. The ideal implant should present a surface conducive to bone growth or that will induce osseointegration, providing a direct structural and functional connection between ordered living bone and the surface of a load-carrying implant, regardless of implantation site, bone quantity, bone quality, etc. The surface of HA is highly bioactive and can stimulate bone to bond with it, and it can thus be said to be osseointegrative [20]. HA particles are exposed on the surface of SLS sintered articles and thus may be able to provide the functions of bioactivity and osseointegrativity. Further analysis is needed to confirm these functions. The large degree of HA exposure with the use of compounded matrix composite materials is in contrast to the results of a study using physically blended materials. A previous study of the SLS sintering of PEEK–HA composite blends by Tan *et al.* [13] resulted in the HA particles being only partially exposed. In addition, the HA particles used in the present study had a median size of $3.80\ \mu\text{m}$, and extensive exposure of particles of such size provided the large surface areas to facilitate osseointegrative

functions. For the blended HA composite study by Tan *et al.* [13], the size of the HA particles was 10–50 μm and provided less surface area exposure compared with that provided by the smaller HA particles.

3.4 Effects of the HA content on the laser sintering of HA–HDPE

As shown in Fig. 7, composites with 30 vol % HA produced a thicker layer than composites with 40 vol % HA for the same processing parameters. At 2.4 W, composite powders with 30 vol % HA were successfully sintered, while composite powders with 40 vol % HA were not sintered. This finding indicates that the lower HDPE content of the composite powder (higher HA content) resulted in lesser sintering owing to the lower binder content. Figure 8 shows the surface morphology of a single sintered layer with 40 vol % HA at laser powers of 3.6 and 9.6 W. Similarly to the sintered layer with composite powders of

Fig. 7 Effect of HA content on the height of sintered single layers at a laser scanning speed of 1200 mm/s

30 vol % HA, as shown in Fig. 5, the sintered layer with powders of 40 vol % HA exhibited more pores at low laser power [Fig. 8 (a)] and became denser at high laser power [Fig. 8(b)].

3.5 Specimen building and cross-section analysis

A scanning speed of 1200 mm/s, and laser powers of 3.6, 4.8, 7.2, and 9.6 W were selected to build block specimens of five layers with a 0.4 mm layer thickness. Under all these parameters, the blocks were built with varying degrees of success using powders with both 30 and 40 vol % HA. At 3.6 and 4.8 W, the block remained white, the same as the original powder. At 7.2 and 9.6 W, the colour of the sample became darker, indicating that some degradation had occurred. However, the layers still bonded. Figures 9(a) and (b) show cross-sections of the 30 vol % HA laser-sintered block. A number of pores were observed from the cross-section of the block, indicating that pores existed inside the block and might be interconnected.

At a laser power of 4.8 W, a block with 40 vol % HA could be built and pores existed within it [Fig. 9(c)], but delamination was noted between the layers, as shown in Fig. 9(d). Delamination was also observed in blocks with the composite of 40 vol % HA powder sintered at other power densities. This delamination suggests that the fusion between the layers with composite powders of 40 vol % HA was not as good as that with composite powders of 30 vol % HA owing to the lower HDPE content. The fusion between the laser-sintered layers depends on the depth of the laser-sintered layer and the layer thickness of the deposited loose powder. The depth of the laser-sintered layer should be higher than the layer thickness of the deposited loose powder to allow an overlap between the newly sintered layer and the previous layer, thereby building a fusion interface

Fig. 8 SEM micrograph of sintered single-layer surfaces with the 40 vol % HA composite and a laser power of (a) 3.6 W and (b) 9.6 W at a scanning speed of 1200 mm/s

not enclosed at certain processing parameters under which the particles were partially fused, and thus the pores between them would be interconnected. Future work will be conducted to characterize the open porosity and interconnectivity and optimize the SLS processing to obtain the suitable porosity by adjusting processing parameters or the characteristics of powders to produce tissue scaffolds.

4 CONCLUSIONS

The results presented in this work show that SLS is not only capable of building HA–HDPE products but also brings about necessary features for their application as bioactive implants and tissue scaffolds.

The laser power and scanning speed were found to have a significant effect on the sintering of HA–HDPE composites and influence the degree of fusion between the particles and the porosity existing in the composite layers. Control over the structure and its porosity could be achieved by varying processing parameters. Besides laser power and scanning speed, a number of other processing parameters can also be adjusted and may further control the features and extent of the pores and therefore would have great potential to optimize pore size in HA–HDPE for tissue scaffolds.

In addition, SLS enabled a good number of HA particles to be exposed on the surface of HA–HDPE composites and may provide the products with a higher level of bioactivity. The compounded HA–HDPE particles used in this study allow the HA embedded on the composite surface to be exposed following the surface melting induced by laser irradiation. The exposure of the HA particles would provide bioactivity and osseointegration to the SLS-fabricated implants or tissue scaffolds. Moreover, the HA particles exposed are generally small (median size of 3.80 μm) and provide large bioactive surface areas.

Under the same processing parameters, a lesser degree of sintering was observed with less HDPE (higher HA content) in the composite powders owing to the lower binder content. Since HA plays the bioactive function in the composite parts, this could compromise the effects of the HA content on the sintering and its functions of bioactivity.

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