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Bioinert ceramics scaffolds for bone tissue engineering by laser-based powder bed fusion: a preliminary review

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Abstract. The implementation of laser powder bed fusion (PBF-LB) on ceramics is far more demanding than their metallic and polymeric counterparts for bone tissue engineering (BTE). The review will shed light on bioinert ceramics-based biomaterials manufacturing through PBF-LB incorporating alumina and yttria-stabilized zirconia as oxide-based ceramics and nitride-based ceramics as non-oxide-based ceramics with particular prominence on their properties and requirements for biomedical devices and BTE. The review paper will also classify bioinert scaffolds processed through PBF-LB as a medium to manufacture drug delivery systems (DDS) and to ameliorate critical-sized bone defects based on the fracture site length of the bone with the various modes of functionalization through the incorporation of drugs, stem cells, and growth factors for personalized medicine.

Keywords: bioinert ceramics; bone tissue engineering; critical-sized defects; digital manufacturing; laser powder bed fusion; drug delivery systems;

1. Introduction

Osseous tissue, widely known as bone, provides structural integrity to the living being and has a profound role in shielding the organs, biomechanical movement, and anchoring several blood cells. Bone tissue presents a hierarchy at the macro, micro, and nano-level with the schematic representation depicted in Figure 1. Bone tissue has a peculiar property to dynamically heal and remodel itself from micro-level damages by the activity of osteoblasts and osteoclasts. However, critical orthopedic injuries cannot heal by themselves, and henceforth usually needs engineered structures i.e. biomaterials [1].

Biomaterials can be elucidated as natural or synthetic supporting structures which can assist biomechanical movements. Biomaterials are made of assorted types of materials encompassing metals, ceramics, and polymers. Metallic biomaterials orchestrate and severe for long periods of time without any significant rejection with the characteristics like high corrosion and wear resistance, suited mechanical properties for large bone defects, and biocompatibility. However, the several downsides to using metallic biomaterials which include stress shielding [2] of the mangled bone, bioactivity, and revision of the surgery at the orthopedic defect. On the contrary, polymeric biomaterials have the largest batch of materials used in hard tissue restoration but the disadvantages of the polymeric materials include lower elastic modulus and tensile strength, time-dependent degradation based on the complexity of the polymeric chains, and, maintenance after the sterilization before surgery [4].

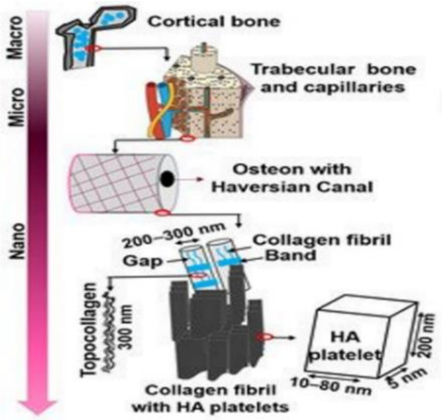

Figure 1. The bone hierarchy at the macro, micro, and nano-level, adapted from [3]

Ceramic biomaterials have peculiar attributes and present several advantages which can fulfill the shortcomings and gaps of metallic and polymeric biomaterials since ceramics being inorganic compounds are made up of metals and nonmetals linked by ionic and/or covalent bonds with forming crystal structures. The bestowed property on ceramics imparts them crystallographic and texturing properties coupled with chemical functionalization of the ceramics to fine-tune the degradation and bioactivity with the physiological medium making them excellent candidates for BTE. Ceramic biomaterials or bio-ceramics can be broadly classified into three different categories which include bioactive, bioresorbable, and bioinert ceramics. Bioactive and bioresorbable ceramics have the potential to bond with the bone tissue in the vicinity due to the similar inorganic composition of bone through the nucleation [5,6] and crystal growth of hydroxyapatite (HA) [7]. Therefore, there is a chemical reaction between the bone tissue and bioactive and bioresorable ceramics. On the other hand, bioinert ceramics remains inert and therefore there is no chemical reaction between the surrounding defective bone tissue and bioinert ceramics since the chemical composition of the bioinert ceramics is different from the bone. Most of the articles in the literature are devoted to bioactive and bioresorbable ceramic scaffolds. However, on the contrary, there is a single review [8] devoted to bioinert ceramics which was our prime motivation for writing the present review article to fulfill the gap in the literature.

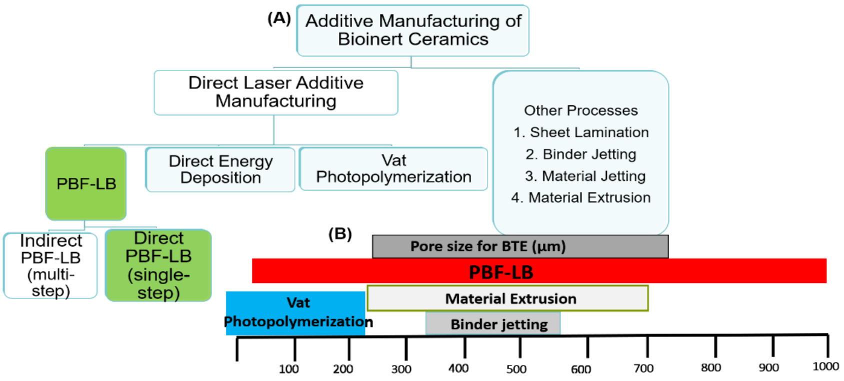

Figure 2. (A) Current additive manufacturing methods for bioinert ceramics scaffolds and process categories highlighted in green are the ones discussed in this review paper, (B) Advantage of PBF-LB over other AM techniques used for BTE, adapted from [3]

The additive manufacturing (AM) of bioinert ceramics scaffolds encloses techniques like PBF-LB, material extrusion, directed energy deposition, binder jetting, vat-photopolymerization, sheet lamination, and material jetting as depicted in Figure 2(a). However, the most commonly used techniques from Figure 2(a) for bioinert scaffold manufacturing are PBF-LB, material extrusion, binder jetting, and vat-photopolymerization. Amongst all the commonly used techniques used for BTE, PBF-LB has a major advantage in the fabrication of the scaffolds since it presents a wider window for the fabrication of pore size of scaffolds ranging from 100 – 1000 µm as shown in Figure 2(b). The functionality of scaffold manufactured by PBF-LB can further be enhanced by the incorporation of hydrogels [9]. They have the characteristics of absorbing and retaining water into their structure (20–100% water relative to the total mass), without dissolution of the material into the pores of the scaffold. Secondly, the most favored way of enhancing the functionality of the scaffolds is the incorporation of DDS on the powder bed for personalized medicine. DDS encompasses the incorporation of a drug, an active molecule, and cells in the scaffolds fabricated by PBF-LB with the more detailed information presented in section 4. Additionally, PBF-LB has the capability to manufacture customized shapes to fulfill the defect at the anatomical site of the defective bone based on the critical size defects. Therefore, taking the advantage of PBF-LB process as the most applicable AM technique for bioinert ceramics from the perspective of BTE is the second motivation to write the review article.

The authors have described the manufacturing of bioactive ceramic scaffolds for BTE through the PBF-LB technique [3]. The review article sheds light on silicate-based ceramics, hydroxyapatite, and bioactive glass-based ceramics processed through direct PBF-LB. However, as far as bioinert ceramics scaffolds the number of articles published in the last 13 years are depicted in Figure 3.

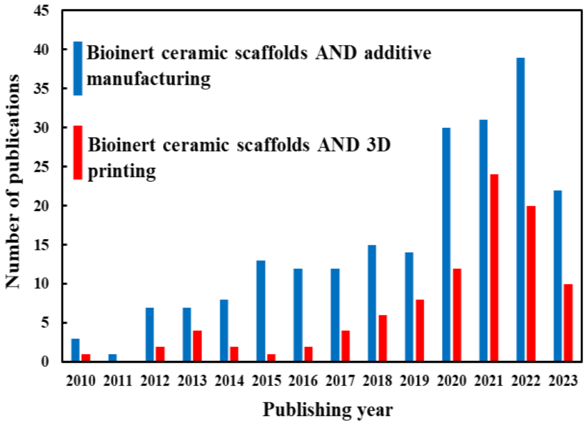

Figure 3. The number of articles published in the last 13 with different keywords years

Two different sets of keywords are used as shown in Figure 3. Furthermore, there was not even a single review article in Scopus with the keywords “Bioinert ceramics scaffolds” AND “laser powder bed fusion” Henceforth, this was our prime motivation to write the review article. This review article will only review direct PBF-LB articles without any use of binders which were used to manufacture bioinert ceramic scaffolds since the scaffolds were not subjected to any geometrical constraints, no additional post-processing treatment like debinding and firing process, single-step technology, unlike most in indirect PBF-LB process of bioinert ceramic scaffolds [10].

The aim of this study is to review the lasers used to consolidate bioinert ceramics materials which are mostly used for orthopedic load-bearing devices (spinal defects, bone defects, and femoral head fabrication) in the form of oxides ceramics as Al_2O_3, and YSZ (yttria-stabilized zirconia), and non-oxides as Si_3N_4 manufactured by direct PBF-LB. Lastly, the manufacturing of bioinert ceramic scaffolds for critical-sized defects of the bone defects ranging from 1-4.5 cm and DDS through PBF-LB are presented.

2. PBF-LB of bioinert ceramics

PBF-LB process is one of the AM techniques as it is used to manufacture the scaffold as shown in Figure 4(a), from a Computer Aided Design/Manufacturing (CAD/CAM). The powder particles on the powder bed are laser melted or laser sintered. The binding mechanism unraveling in the PBF-LB process comprises solid state sintering (Al_2O_3, YSZ), liquid phase sintering/partial melting (Al_2O_3, YSZ), full melting (Al_2O_3), and chemically induced binding (Si_3N_4). However, the process of manufacturing bioinert ceramic coupons through PBF-LB is far more challenging since ceramics usually have higher melting point, brittleness (eventually no plasticity), and poor thermal shock resistance. Furthermore, a rapid heating and cooling process in powder bed results in cracking, and unrequired porosity on the scaffolds [11].

The process parameters of PBF-LB have a profound effect on the functionality of the manufactured scaffolds in-terms of porosity and surface roughness. The main process parameters of PBF-LB for the manufacturing of bioinert ceramic scaffolds encircles around the source of lasing media, the wavelength of laser beam, laser power, laser scanning speed, laser scanning strategy, particle shape and size in the powder bed, and layer thickness as shown in Figure 4(b). Nowadays, ceramic machines are usually implemented through two different lasers CO_2 (wavelength is 10.6 μm) and Nd:YAG (λ:1.064 μm) for the manufacturing of ceramic scaffolds. Furthermore, some ceramic machines in the industry for manufacturing ceramic scaffolds are equipped with both CO_2 (for pre-heating the ceramic powders) and Nd:YAG lasers (for consolidation). Laser adsorption for the oxide bioinert ceramics can be different for both the CO_2 and Nd: YAG lasers. Besides adsorption, laser beam diameter plays a significant role in the amount of energy transferred to the ceramic powder bed and can be expressed as

$$E=P_{eff}/(V_s \cdot h_d \cdot d) \tag{1}$$

where E is the laser energy density, P_{eff} is the laser power, V_s is the laser beam scanning speed, h_d is the hatch distance and d is the layer thickness. Higher scanning speed will lead to poor sintering/melting of the layer and vice versa. Also, authors have shown in recent studies that modulating the scanning speed can eventually fine-tune or customize the desired phases in the ceramic scaffold [12].

Particle morphology also has a perspicacious effect on the final microstructure of the manufactured scaffold as they are directly related to the packing density of the powder bed and flowability. As the basic criterion and preferred AM norm is the powder of narrow particle size distribution with a spherical size distribution, the better will be the flowability. The advantages of using multimodal powders as the feedstock since the packing bimodal size distribution can fulfill the interstitial sites/voids of the powder bed. The preferable coarse-to-fine particle size ratio is (1:10) with the weight fraction of the coarser particles being 70% to accomplish maximum packing [10]. Therefore, all the parameters depicted in Figure 4(b), of the PBF-LB have direct repercussions on the printability of the scaffold which can have an influence on performance customization of the scaffold.

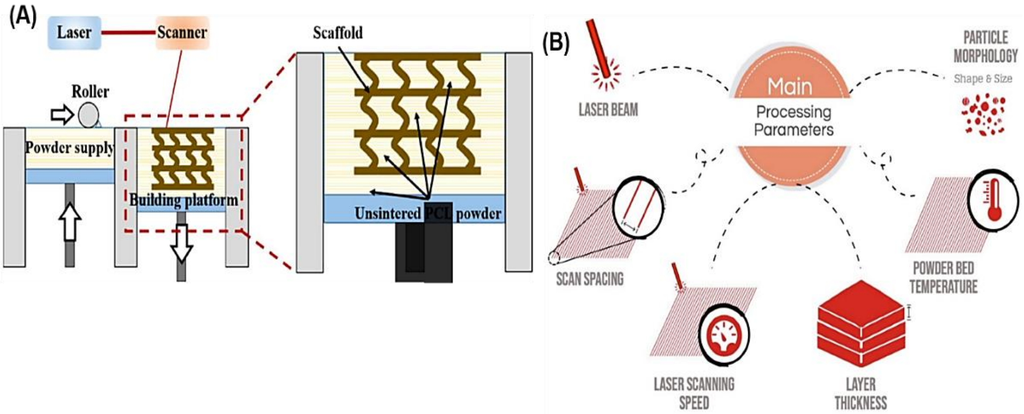

Figure 4. (a) Schematic of PBF-LB process, (b) parameters to be taken into consideration for PBF-LB process, adapted from [13,14]

3. Bioinert ceramic scaffolds processed by PBF-LB

Mostly, the applicability of bioinert ceramics concerning BTE can be on joint prostheses, spine fixtures, and femoral head manufacturing. The most frequently used oxide bioinert ceramics are alumina, YSZ, and non-oxide bioinert ceramics including Si_3N_4. They offer several advantages like less oxidation and corrosion resistance in the body fluids along with low levels of friction and wear. However, on the downside, they are too brittle with low elasticity. The materials i.e. alumina, YSZ, and Si_3N_4 will be presented in detail and are processed through direct PBF-LB.

3.1 Alumina

Alumina is a bioceramic used as a potential alternative to metal implants for a total hip prosthesis (load-bearing applications) [15]. The rationale for using alumina for load-bearing applications can be attributed to the low friction coefficient, high hardness and abrasiveness, chemical inertness, and good thermal conductivity which makes alumina a potential candidate for BTE [16]. It has been also proven from the clinical point of view that the alumina-on-alumina bioceramic on the femoral with the acetabular side leads to minimal osteolysis, which can be associated with superior wear resistance [17,18].

However, alumina has a drawback concerning BTE. It is limited to the capability of the formation of non-adherent fibrous mass or membrane at the bio-interphase when implemented as the implant material. As a result, it can lead to implant failure at the bio-interphase [19]. Nevertheless, the alumina implant failure at the bio-interphase can be averted by removing surface defects (surface modification) or even modifying the surface features at the nano-scale to reduce the pro-inflammatory macrophage response [20]. All these aforesaid surface modifications could facilitate the strong interfacial bonding between alumina and defected bone and can be prevented from loosening [21].

The first and foremost implementation of alumina ceramics by PBF-LB dates back to 1995 when Subramanian et al [22] first implemented alumina in the powder feedstock. Various researchers have used various additives for alumina bioceramic processing. The additives include diboron trioxide [23], zirconia [24], and polyamide [25] with the summary of alumina bioinert ceramics shown in Table 1.

Table 1. Summary of PBF-LB of alumina bioinert ceramics.

Powder feedstock	**Laser type**	**Scanning speed**	**Reference**
Al_2O_3 (80 wt%)+ stearic acid	Nd:YAG laser	2-60 mm/s	[26]
Al_2O_3 + 6YSZ	CO_2 laser for pre-heating, Nd:YAG laser for sintering/melting	200 mm/s	[24]
Al_2O_3 + 6YSZ	CO_2 laser for pre-heating, Nd:YAG laser for sintering/melting	200 mm/s	[27]
Al_2O_3 + polyamide	CO_2 laser	60 mm/s	[25]
Pure Al_2O_3	Fibre laser	30-1000 mm/s	[28]
Al_2O_3 + additives (Na_2O + Fe_2O_3 + SiO_2 + MgO + TiO_2 + CaO)	Fibre laser	90 mm/s	[29]
Compressed Al_2O_3 powder at different compressive pressure (0, 50, 100 MPa)	CO_2 laser	50-1500 mm/s	[30]

As table 1 illustrates, most of the studies focused on CO_2 and Nd:YAG laser for laser sintering/melting of Al_2O_3. Porous alumina can expedite the bone regeneration and healing process by presenting a non-toxic surface at the bio-interphase [16]. Also, the superior mechanical properties of alumina make it a suitable implant for both open defects and long bone defects (load-bearing application) [31].

3.2 YSZ

YSZ (ZrO_2 - Y_2O_3) has been chiefly used for orthopedic implants/scaffolds [32]. The attractive properties, which make it suitable for orthopedic (hip, knee) encircles around good wear resistance, biocompatibility superior mechanical properties especially fracture toughness, and durability [33]. 3 mol% yttria, the most common and stabilized biomedical grade zirconia possess superior fracture toughness to be used for BTE. YSZ was used for the first time in 1990 to manufacture the femoral head for load-bearing applications. This material was in practice for the femoral head until steady fractures were reported and suggested by the authors in their study [34]. The catastrophic failure was not attributed to the mechanical properties but instead to the phase transformation from tetragonal to monoclinic zirconia [35]. Also, the low thermal degradation by occupying the anionic variances (oxygen) in water molecules, or called ageing could also evoke this catastrophic transformation in zirconia. Alumina-reinforced or toughened zirconia and cerium oxide incorporation have comparatively shown better and enhanced ageing resistance when compared to the YSZ counterpart.

PBF-LB of zirconia and zirconia toughened ceramics are also used for dental implants apart from the load-bearing bone implants and are explored by various researchers as it depends on the limited processing steps (no debinding stage) with various examples by the researchers in Table 2 of direct PBF-LB process on YSZ.

Table 2. Summary of PBF-LB of YSZ bioinert ceramics.

Powder feedstock	**Laser type**	**Scanning speed**	**Reference**
ATZ (Alumina (80 wt%) toughened zirconia (20 wt%))	Nd:YAG laser	200 mm/s	[36]
YSZ (ZrO_2-8Y_2O_3)	Nd:YAG laser	60-75 mm/s	[37]
Eutectic mixing ratio of 41.5 wt% YSZ and 58.5 wt% of Al_2O_3	CO_2 laser for pre-heating, Nd:YAG laser for sintering/melting	200 mm/s	[24]
ZrO_2 (90 wt%) + Y_2O_3 (10 wt%)	Nd:YAG laser	2-60 mm/s	[26]
ZYP30 (Zircar)	Fibre laser	1250-2000 mm/s	[38]
ZrO_2 – Al_2O_3 various blends	CO_2 laser for pre-heating, Nd:YAG laser for sintering/melting	200 mm/s	[27]

As Table 2 illustrates the majority of the studies focused on CO_2 and Nd:YAG laser for laser sintering/melting of YSZ. The limitation of zirconia concerning laser interaction is very limited due to the unstable thermal behavior of zirconia, which can induce crack formation and ultimately lead to the failure of the implant. One way of avoiding the cracks is introducing a dual laser system (CO_2 and Nd:YAG) operating at different wavelengths. The formation of the cracks can be avoided by heating the powder bed with a CO_2 laser operating at a longer wavelength and shorter frequency (CO_2 laser) and followed by melting the ceramic powder with Nd:YAG operating at a shorter wavelength and higher frequency to fabricate the bioceramic dense part without any cracks and superior strength.

3.3 Silicon nitride

Silicon nitride ceramics are admired for their corrosion resistance, hardness, fracture toughness, and wear performance. The lifetime of the oxide ceramics (Al_2O_3, YSZ) is limited due to its oxygen activity in the simulating body fluids (hydrothermal environment). However, on the contrary, Si_3N_4 has a high wear rate initially followed by the formation of hydroxylated silicon oxide will be formed as the primary product of the thermomechanical wear and resulting in decreasing friction level which is a mandate for high-load medical applications [39].

Si_3N_4 also complies well with biomedical applications such as dental and orthopedic implants/scaffolds. Si_3N_4 meets the satisfactory requirements of orthopedic surgery displaying several attractive features like low solubility in the aqueous solutions, high wear resistance, and proper mechanical properties for BTE. The other complemented properties with regards to BTE include anti-bacterial properties, biocompatibility, and excellent radiographic imaging, thus making them strong candidates to support high loads for deformed or mangled bone. The specific targets of Si_3N_4 as the biomaterial focuses on the locations like intervertebral spacers, spinal surgery, joint arthroplasty, otorhinolaryngology, dental, osteo-fixation system, and orthopedic scaffolds [40]. Table 3 shows the summary of the direct PBF-LB on Si_3N_4. As Table 3 shows scarce studies have been devoted to PBF-LB of Si_3N_4 ceramics since the binding mechanism type is called chemically induced binding which tends to dissociate Si_3N_4 ceramics.

Table 3. Summary of PBF-LB of Si_3N_4 bioinert ceramics.

Powder feedstock	Laser type	Scanning speed	Reference
Si_3N_4 hollow microspheres	CO_2 laser	150 mm/s	[41]
Si_3N_4 powder consolidation on pure titanium	Nd:YAG laser	pulse time 1-20 ms	[42]
Silicon powder as the powder bed and further nitriding the green part	Nd:YAG laser	80 mm/s	[43]

4. Manufacturing of bioinert ceramic scaffolds for critical-sized defects and drug delivery system through PBF-LB

Critical-sized bone defects, commonly with a length irrespective of the dimensions and usually in the range of 1-4.5 cm can be repaired by synthetic bioinert grafts/scaffolds. The scaffolds which can be implemented on bone defects encompass the femur, shoulder, hip, wrist, tibia, ankle, and spinal cord. Synthetic bioinert bone scaffolds play an imperative role in BTE manufactured by PBF-LB by repairing spinal defects, bone defects, femoral head fabrication, and heart valves by facilitating and providing a 3D network for regenerating the bone for critical defects. PBF-LB process can be also used as a DDS [44] as shown in Figure 5. In formulations, I and II, the drug and active biomolecules can be directly blended with a powder bed of ceramic powders. However, in these types of formulations, the active ingredient is directly subjected to the thermal energy imposed by laser which can degrade them or destroy the functionality of the drug or active biomolecules. In order to diverge from the aforementioned formulations, III and IV types of formulations can be implemented on the powder bed by necessitating the incorporation of the drug molecules by surface functionalization onto the bioinert scaffolds. Henceforth, PBF-LB process presents a very flexible approach to manufacturing orthobiologic materials as a mediator to deliver drug or active biomolecules. In recent studies, authors proved that manipulating the scan spacing can fine-tune drug release in the in-vitro conditions by PBF-LB process [45].

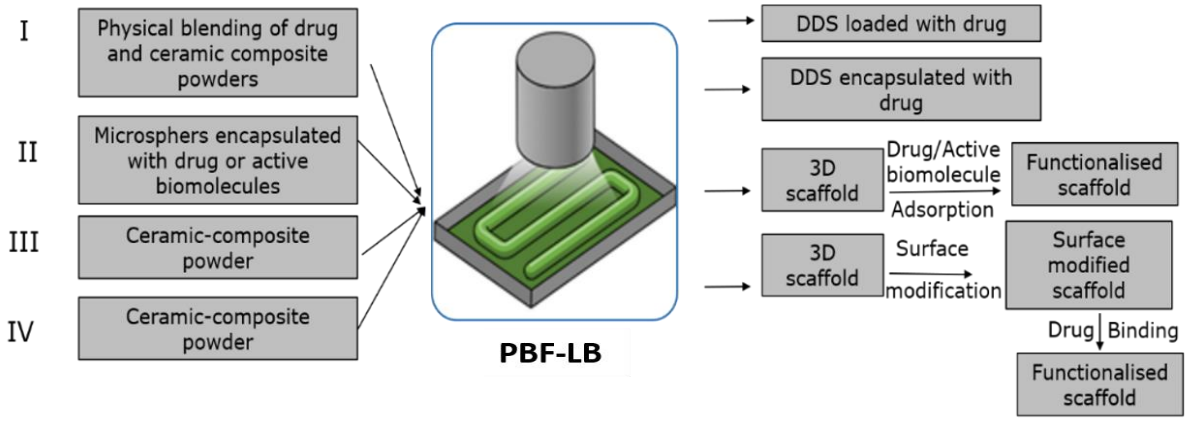

Figure 5. Schematic of developing DDS through PBF-LB process

The first class of synthetic bioinert scaffolds which can affect the functionality PBF-LB allows custom-designed bioinert scaffolds suitable to the defect by having superior control over pore size, porosity, and surface roughness. The second class of synthetic bone scaffolds manufactured by PBF-LB can also be integrated with active biomolecules to have DDS directly on the powder bed. The second class of bioinert scaffolds is mainly used for cervical and lumbar spine, wrist, ankle, and grim fractures of the femur and tibia, where the fracture size is between 2 to 4 cm. The third and the last class of bioinert ceramic scaffolds are usually implemented in the femoral neck fracture of the hip joints and joint arthroplasty by imparting functionality through stem cells by embodying onto the scaffold as shown in Figure 6.

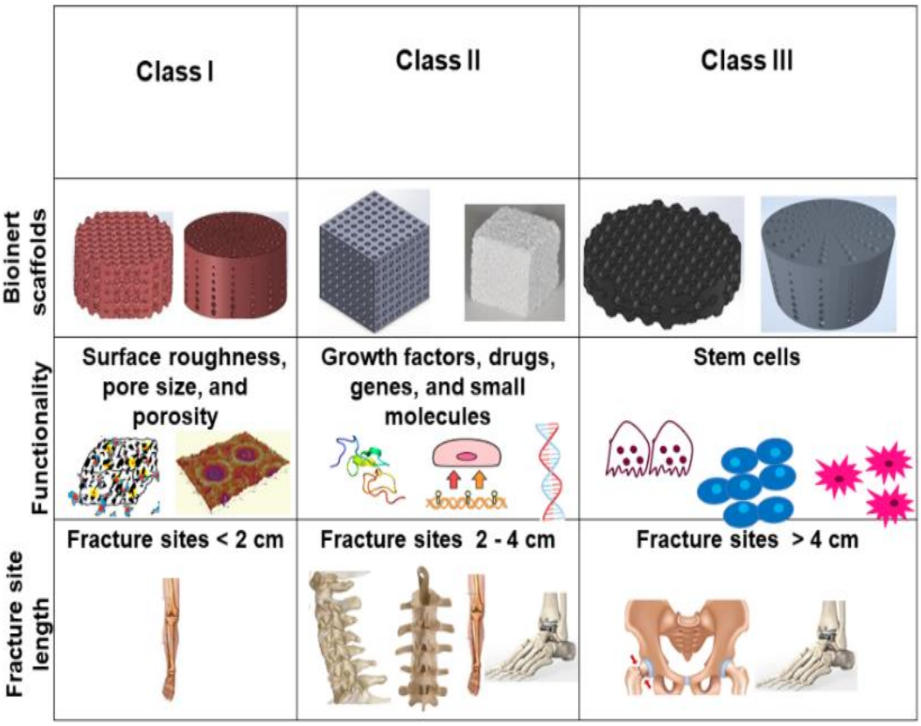

Figure 6. Three major classes to enhance the functionality of the bioinert ceramic scaffolds for bone repair by the PBF-LB process, adapted from [3]

5. Discussion and potential developments

Ideally, it is a very strenuous task to fabricate an ideal scaffold, which can fit both the dense and the porous parts of the bone. One solution can be to fabricate a triple periodic minimal surface (TPMS) by improving the architectural design through ceramic materials to withstand mechanical loads and significantly increase the permeability of body fluids. The metal porous TPMS can be easily fabricated and are well known for BTE [46]. However, on the contrary, there is very limited progress and development of TPMS bioceramics surface, although there is a recent study existing in the literature. Furthermore, recently, ceramics powders are incorporated into metal lattices by consolidating through spark plasma sintering [47] for biomedical applications [48].

Usually, bioceramics are not well suited for shorter wavelengths and higher frequencies. A probable solution can be the mixing of the additive with a bioceramic powder bed. The additive itself should be good orthobiologic material that can coalesce actual ceramic particles in the powder bed to sinter or melt. For instance, silicon can act as an efficient absorber of the fiber laser to manufacture the scaffold for drug delivery [49]. The future recommendation of PBF-LB for ceramics can be focused on the customized machine laser designs and process parameters.

6. Conclusions

In this review, we presented the different ceramic materials used to produce bioinert scaffolds through PBF-LB. Alumina and YSZ are well versed with PBF-LB but on the contrary Si_3N_4 ceramics have to be studied more by the PBF-LB process. Si_3N_4 ceramics have a binding mechanism type called chemically induced binding which tends to dissociate Si_3N_4 ceramics. One solution to tackle this problem would be to have an over-pressure of nitrogen in the powder bed atmosphere in order to prevent the dissociation of Si_3N_4. Henceforth, this area can also be explored in further studies. Furthermore, the most used lasers for bioinert ceramics are CO_2 and Nd:YAG lasers with a very limited number of studies on fibre lasers. This can be attributed to the fact that fibre lasers are more expensive than the CO_2 and Nd:YAG lasers which are used in bioinert ceramics. Finally, it was presented that PBF-LB has the potential to fabricate scaffolds by incorporating drugs, and biomolecules straightway in the powder bed. The customized scaffolds fabricated through PBF-LB can be a solution for personalized medicine for bone repair along with gateways to DDS.

Credit authorship contribution statement

N Kamboj - Writing – review & editing, Visualization, Investigation, Answering reviewer comments. **H Piili, A Ganvir, A Gopaluni, C Nayak, N Motiz, A Salminen** – Editing, Visualization, Investigation.

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