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RESEARCH ARTICLE | DECEMBER 10 2020

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Special Collection: Special Topic Collection: Biointerface Science in Australia 2020 — An issue in celebration of Hans Griesser's career

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Biointerphases 15, 068501 (2020)
https://doi.org/10.1116/6.0000557

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Biointerphases
A Journal of Biomaterials and Biological Interfaces

PERSPECTIVE

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Cite as: Biointerphases 15, 068501 (2020); doi: 10.1116/6.0000557
Submitted: 16 August 2020 · Accepted: 11 November 2020 ·
Published Online: 10 December 2020

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Note: This paper is part of the Biointerphases Special Topic Collection on Biointerface Science in Australia 2020 - An issue in celebration of Hans Griesser's career.

ABSTRACT

Additive manufacturing (AM) of orthopedic implants is growing in popularity as it offers almost complete design flexibility and freedom, meaning complex geometries mimicking specific body parts can be easily produced. Novel composite materials with optimized functionalities present opportunities for 3D printing osteoconductive implants with desirable mechanical properties. Standard metals for bone implants, such as titanium and its alloys, are durable and nontoxic but lack bioactivity. Bioactive glasses promote strong bone formation but are susceptible to brittle failure. Metal-bioactive glass composites, however, may combine the mechanical reliability of metals with the bone-bonding ability of bioactive glasses, potentially reducing the incidence of implant failure. Processing such composites by AM paves the way for producing unprecedented bespoke parts with highly porous lattices, whose stiffness can be tailored to meet the mechanical properties of natural bone tissue. This Perspective focuses on titanium-bioactive glass composites, critically discussing their processability by AM and highlighting their potential as a next-generation implantable biomaterial.

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I. INTRODUCTION

New biomaterials are frequently being developed for orthopedic implants capable of integrating with surrounding hard tissues to remedy bone defects and fractures. Currently, there is no straightforward solution to restore the physiological function of damaged or diseased bone, especially in the presence of critical defects incapable of healing spontaneously during the patient's lifetime.[1,2] Current approaches for treating critical bone defects include distraction osteogenesis, vascularized bone grafts, and induced membrane technique.[3,4] However, these methods have substantial shortcomings, including potential infections or malunions.[5] Researchers are faced daily with new clinical problems that initiate the continuous evolution of current remedies and lead to breakthroughs in design, materials development, and manufacturing techniques. New approaches such as patient-specific implant therapy, tissue engineering, and biofabrication are emerging to help improve the patient's health and physical ability. Biomaterials are advancing the cause of orthopedic surgery, with novel bone scaffolds to effectively support tissue repair, stimulate physiological healing processes, and restore load-bearing functions being developed. Such advancements can provide patients with improved bone prostheses that ensure comfort and well-being, with a service lifetime of 15–20 years for the elderly, and more than 20 years for younger generations. In parallel, novel technologies are being developed to redesign the shape and the internal architecture of bone implants to achieve patient-specific geometry and performance. This careful engineering encompassing new materials and new technologies sets the basis for next-generation orthopedic implants, yet there are crucial issues to be resolved prior to standardization in clinical practice. This perspective is specifically dedicated to the critical discussion of the advantages and disadvantages of titanium-bioactive glass composites (Fig. 1), including their additive manufacture and properties, for improved bone scaffolds.

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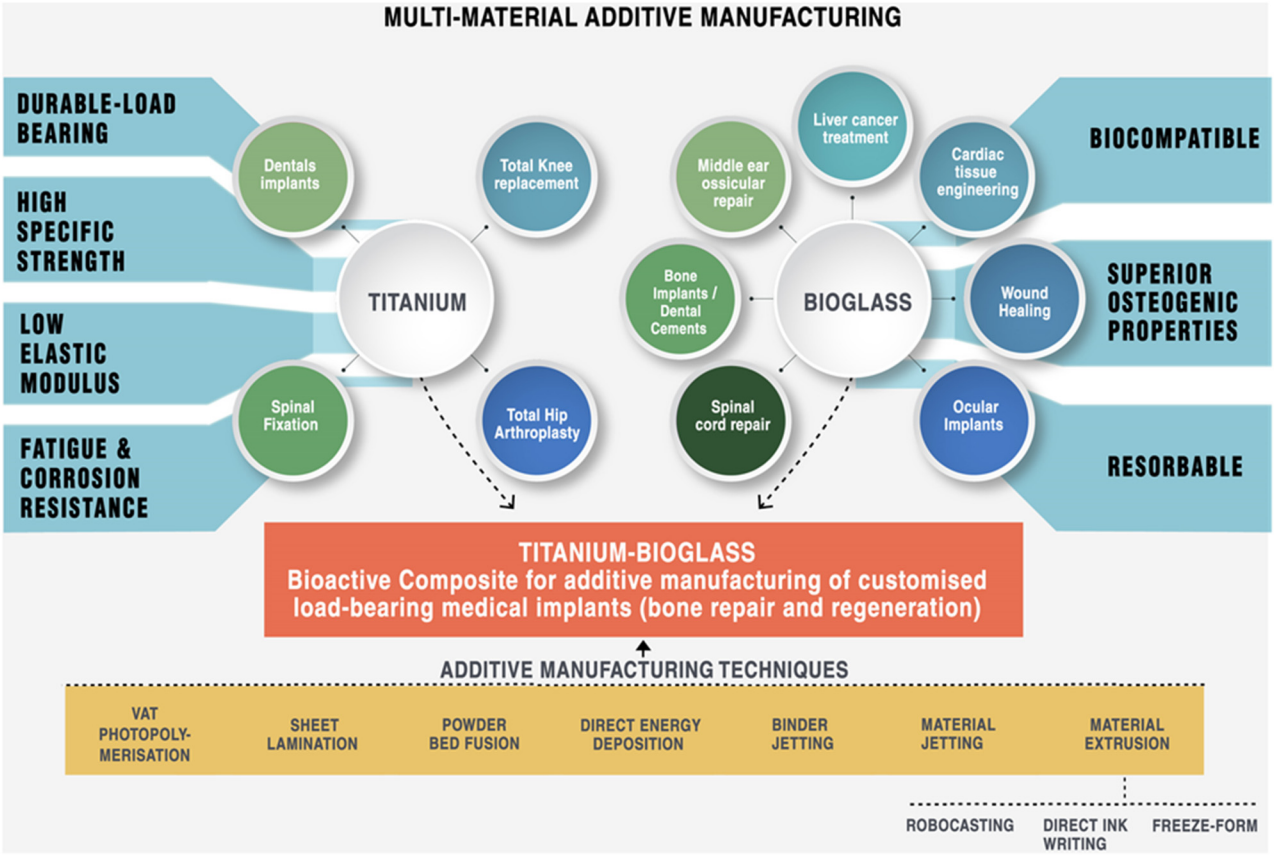


FIG. 1. Schematic detailing the advantages of an additively manufactured titanium-bioglass composite.

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II. TITANIUM-BIOGLASS COMPOSITE: A PROMISING CANDIDATE?

Orthopedic biomaterials should meet specific requirements to qualify for implantation. The essential prerequisite is biocompatibility, achieved by having stable chemical composition to enhance cell proliferation and avoid inflammation.[6] Corrosion and wear resistance ensure implant longevity via excluding the risk of degradation, minimizing debris generation, and avoiding toxicity.[7] Also, orthopedic biomaterials should match the mechanical properties of bone, especially its relatively low modulus and good fracture resistance[8] as implants seek to mimic the natural structure of bone. Consequently, the microstructure of orthopedic biomaterials should be designed to reproduce the porous architecture of bone to induce bone ingrowth and vascularization, as well as to control the implant density and stiffness.[9]

The surface topography guides the cell behavior and kinetics, with direct consequences on the effectiveness of cell adhesion, growth, and the rate of bone healing.[10] Biological interactions mainly occur at the implant-bone interface, where inflammation, allergy, degradation, and loosening are commonly a sequel to failures in biocompatibility.

Current metals of interest in the orthopedic field include medical-grade titanium (Ti) and Ti alloys, especially Ti–6Al–4V [Fig. 2(c)]. Ti is widely used in medical implant manufacturing due to its high specific strength, relatively low elastic modulus, good fatigue and corrosion resistance, and biocompatibility.[15] Ti and Ti alloys are durable and suitable for long-term usage;[16–18] however, Ti and Ti alloys lack of bioactivity causing fibrosis (formation of thin connective tissue around the prosthesis usually conducive to failure). As such, researchers are investigating the addition of new materials such as bioactive glasses, to form new Ti composites.

The bioactive glass, known under the trade-name "45S5 Bioglass®" (composition in oxide weight %: 45% SiO_2, 24.5% CaO, 24.5% Na_2O, 6.0% P_2O_5), was the first artificial biomaterial to exhibit strong bone formation via the release of biologically active ions.[19] Thus, 45S5 Bioglass® has been successfully used as a bioreactive component in many innovative applications, such as cardiac tissue repair, liver cancer treatment and more extensively in bone and dental repair, and reportedly has contributed to the treatment of over a 1.5×10^6 orthopedic cases.[20] 45S5 Bioglass® and its derivatives exist in the market as commercialized products Fig. 2(d) with some compositional differences for specific usage, such as

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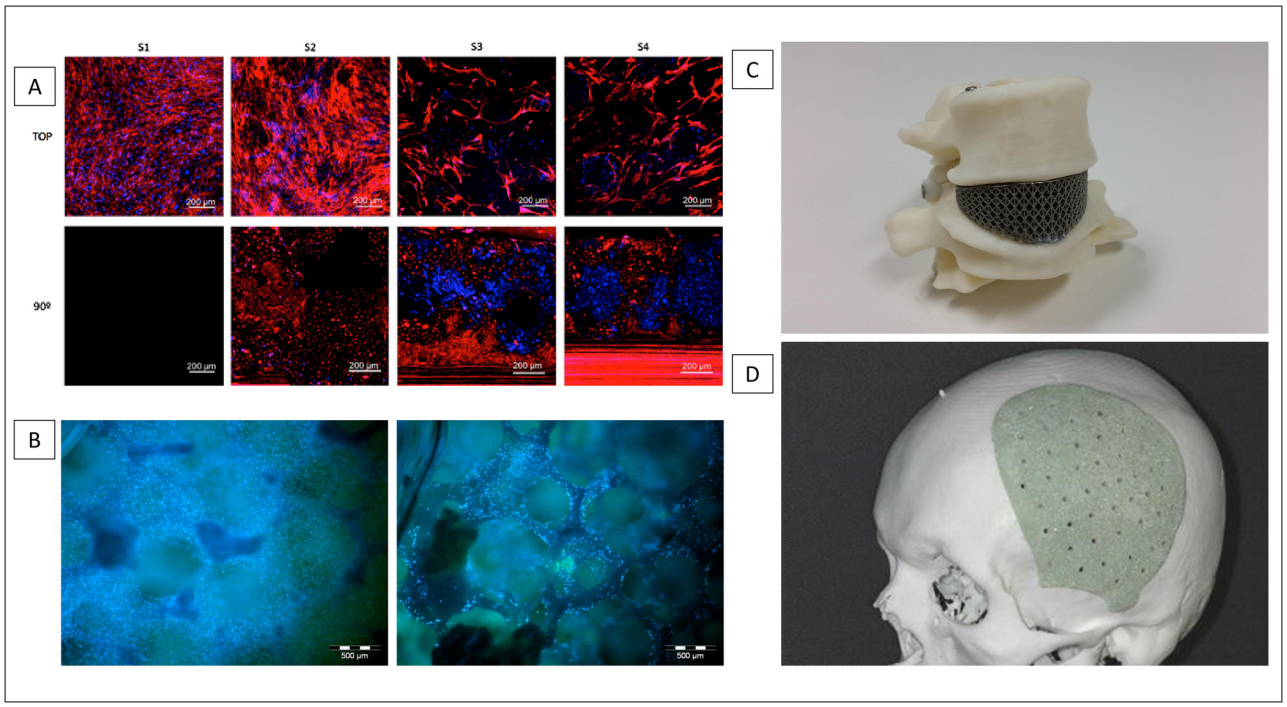


FIG. 2. Example of cell viability on different titanium and Bioglass® surfaces. (a) Fluorescence microscopy images of human mesenchymal stem cells (hMSC) cultured for 7 days on Ti6Al4V cast (S1), Ti6Al4V SLM structures (S2), Ti6Al4V SLM structures with a 2.05 wt. % of bioglass (S3), and with a 2.50 wt. % of bioglass (S4) (Ref. 11). (b) Fluorescence images of 4′,6-diamidino-2-phenylindole (DAPI) stained adMSC (left) and ucMSC (right) in 45S5 Bioglass® based scaffolds after 35 days of cultivation (Ref. 12). (c) A titanium spinal implant cage (Ref. 13). (d) PMMA and bioglass (BoneAlive) implant (Ref. 14). Figure is comprised of multiple reprinted figures. Figure 2(a): Reprinted with permission from Melo-Fonseca *et al*., Mater. Des. **160**, 95 (2018). Copyright 2018, Elsevier. Figure 2(b): Reprinted with permission from Boccaccini *et al*., J. Biomed. Mater. Res. Part A **103**, 1029 (2015). Copyright 2014, John Wiley and Sons. Figure 2(c): Copyright 2015, RMIT University. Figure 2(d) is attributed to Peltola *et al*. (Ref. 14) (CC-BY-NC).

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Novabone (spinal fusion autograph) and BonAlive (craniofacial bone graft).[21]

Figure 2 shows the cell viability on different Titanium Bioglass® surfaces and scaffolds with increased cell numbers reported for Ti6Al4V structures produced by selective laser melting (SLM) with 2.50 wt. % of bioglass.[11,12] Bioactive glass-based devices and scaffolds (where "scaffolds" specifically refers to highly porous materials that are designed to support bone ingrowth and spontaneous healing) possess exceptional osteoinductive and osteogenic properties compared to other ceramic-based implants.[22] Oonishi *et al*.[23] proved that 45S5 Bioglass® was able to induce bone formation more rapidly than either apatite-wollastonite (A-W) glass-ceramic, or synthetic hydroxyapatite [HA, $Ca_{10}(PO_4)_6(OH)_2$], although apatite is the main inorganic constituent of bone and tooth enamel. Further, 45S5 Bioglass® was resorbed more quickly than A-W glass-ceramic, whereas synthetic HA was not resorbed at all.[23] 45S5 Bioglass® improves the implant-tissue bonding strength by inhibiting bacterial film formation by increasing the osmotic pressure and creating an alkaline pH environment by releasing Ca^{2+} ions at the implantation site.[24] Various strategies have been formulated also to induce vascularization through mesoporous bioactive glass-based constructs to support cell viability and tissue in-growth within the scaffold.[25]

45S5 Bioglass® and bioactive glasses, in general, are brittle and unsuitable for load-bearing implant applications. As a result, bioactive glasses are used in advanced coatings for metal implants, where the metal substrate provides mechanical stability and the bioactive glass coating provides the connection to the host tissue. Despite very promising results, the uptake of bioactive glass coatings is hindered by their degradation behavior, since the role of coatings is to allow strong adhesion to host tissue while gradually degrading.[26] Due to their reactivity, bioglass-based coatings are likely to degrade too fast and create areas of coating breakdown, which affects the adhesion of host cells to the implant. Moreover, the material behavior is influenced by the sintering process' temperature and length. Heat-induced crystallization is known to hinder the sintering process and this leads to increased porosity and lower densification in the part, and consequently, degradation in the mechanical properties and poor adhesion at the interface.[27] All in all, this is expected to result in an unstable connection and eventually implant loosening. Other limitations arise from the mismatch in thermal expansion between glass coating and metal implant leading to coating debonding during processing or even after implantation.[28]

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III. ADDITIVE MANUFACTURING OF TITANIUM-BIOACTIVE GLASS COMPOSITES: THE WHAT, WHY, AND HOW

A. Formidable technological challenge

Additive manufacturing (AM) offers an unparalleled geometric freedom to print bone-mimicking complex parts. Additionally, AM provides the opportunity to print parts with superior mechanical properties. The fabrication of fine microstructures contributes to enhancing the fatigue behavior of AM Ti in comparison to traditional fabrication methods.[29] AM Ti parts have also exhibited higher wear resistance in comparison to as-cast parts.[30] Postprocessing of AM parts via hot isostatic pressing (HIP) aims at refining the microstructure further and reducing the lack of fusions. Surface treatment is also a necessity to enhance the tensile properties of AM parts.[31]

There is a certain specificity in AM in which some AM techniques are appropriate for some materials, but not for others, and vice versa. A technological challenge is the identification of an AM technique that is suitable for both metal and glass feedstocks. Ceramics are mainly processed via robocasting, in the form of binder-assisted slurry or of molten material, or via binder jetting, in the form of powder. Glass and ceramic scaffolds fabricated via robocasting are usually printed in an oil bath to reduce the drying speed and to enhance the layers' adhesion. Super high-performance ceramics with superior surface quality are fabricated by binder jetting to produce near-net shape complex geometries (Fig. 3). Conversely, metal prints are mainly fabricated via laser metal deposition (LMD)[36–39] or by powder bed fusion (PBF) techniques.[40]

Current literature presents the challenges to print metallic and ceramic parts using different AM techniques, such as extrusion based AM (Ref. 41) and binder jetting where support structures and binding materials are needed for successful printing, respectively.[42] However, it is still unclear whether these alternative AM approaches are suitable to process metals and glasses simultaneously. For such reason, this perspective is focused on presenting the opportunities and limitations of coprinting bioglass and titanium as distinct powders via LMD and SLM.

LMD facilitates the layer-by-layer buildup of multimaterial geometries such as the titanium-diamond composite recently developed by Fox *et al.*[43] LMD flexibility in changing the feedstock material allows easier control of the print's microstructure and of its mechanical properties while generating parts with finer

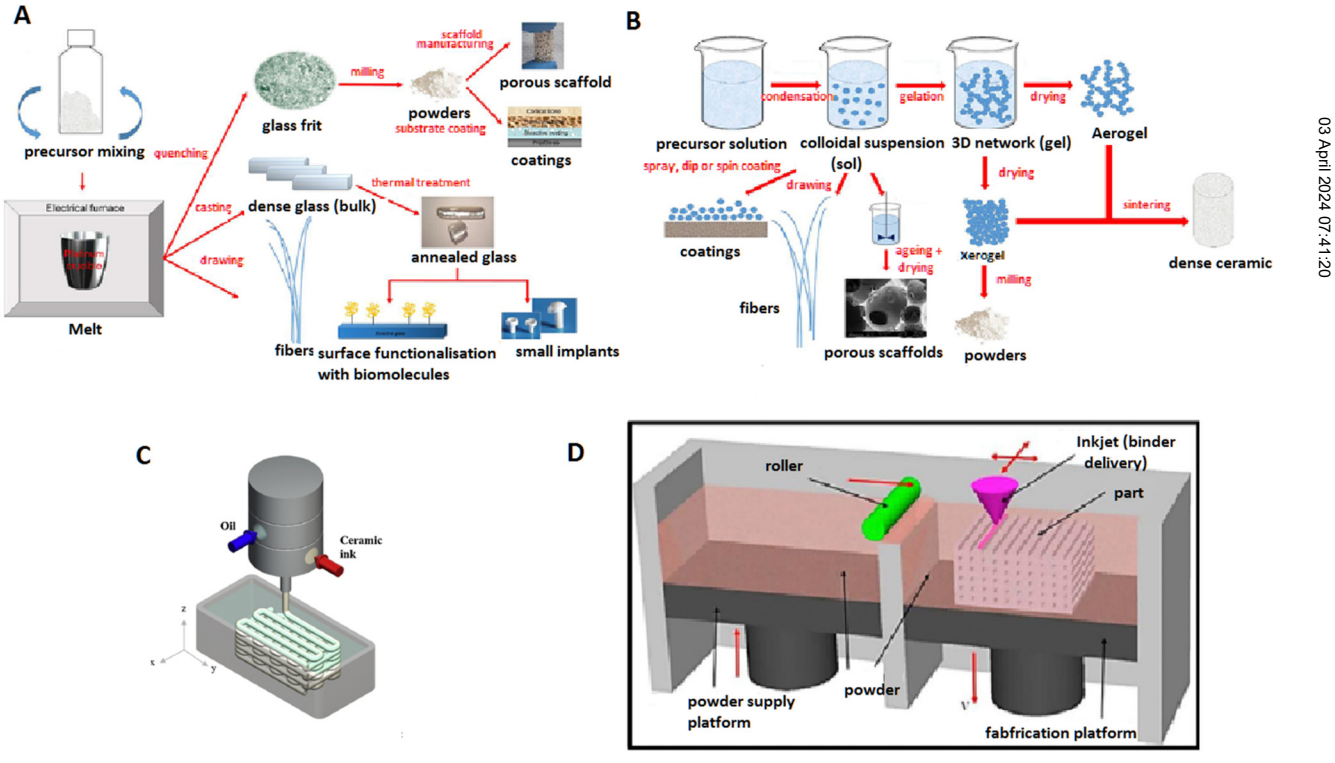


FIG. 3. Schematic illustration of bioglass printing techniques. (A) Bioglass formation through melt-quenching and (b) solgel route (Refs. 32 and 33). (c) Robocasting process of ceramic scaffolds (Ref. 34). (d) Binder jetting where a roller is used to spread powder through the height difference between the powder supply and fabrication platforms and a recoater is used to spread powder from a top hopper using vibration (Ref. 35). Figure 3 is comprised of multiple reprinted figures. Figs. 3(a) and 3(b) are attributed to Kargozar *et al.* (Ref. 32) (CC-BY-4.0). Figure 3(c): Reprinted with permission from Paredes *et al.* Copyright 2019, Elsevier. Figure 3(d): Reprinted with permission from Lv *et al.* Copyright 2019, Elsevier.

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microstructure compared to other AM techniques due to its high cooling rates and steep thermal gradients.[44] Although LMD provides the required flexibility to interchange Ti and glass feedstocks to print layers alternatively, this technique is rarely researched for ceramic-metal composites, as the printing temperature and the total energy should be carefully adjusted. Moreover, the thermal conditions should be monitored closely to minimize glass crystallization phenomena, which are known to reduce the glass bioactivity by lowering its biodegradability.

SLM is a PBF technique suitable to produce complicated shapes. SLM enables processing conditions control ensuring reproducibility, uniformity, and repeatability of printed parts. So far, two basic approaches have been put forward to fabricate composite parts. The "*ex situ*" method starts from two (or potentially more) powders whose composition corresponds to the constituent phases that are desired in the finished part. The powders are premixed and fed into the printer. The composition of the starting phases is expected to remain unchanged upon processing, although the powders get partially or completely molten under the laser beam. The "*in situ*" approach is based instead on controlled reactions that occur upon processing between the precursor powders to achieve new constituent phases. Due to the complicated composition and amorphous structure of bioactive glasses, the "*ex situ*" approach is deemed to be more suitable to fabricate Ti-bioactive glass composites.

At present, LMD and SLM are, therefore, the most appropriate AM techniques to process metal-ceramic composites. For powder-based AM technologies like LMD and SLM, the powder feedstock must meet some basic requirements in terms of particle shape and size distribution in order to be processable. This poses another challenge, since glass powders and metal powders must be fed into the printer within the same job to obtain a composite part, although they usually differ in terms of geometric features. Powders are fabricated in different ways depending on the chemical, microstructural, and physical properties of the material. Ceramics are processed generally by crushing and milling. As an alternative route, ceramics can be produced by the solgel method that results either in a dense ceramic body via sintering or in a very fine powder via milling as the final step. Accordingly, bioactive glass powders can be produced either by melt-quenching and subsequent milling or by solgel method and milling. Solgel powders are usually smoother and more regular than melt-quenched ones, but all particles obtained by milling are generally angular, with corners and sharp edges (Fig. 3). Conversely, Ti powders are atomized [by water, gas, or plasma rotate electrode process (PREP) atomization] to produce spherical particles with a controlled size distribution to be used for AM.[45]

B. To additive manufacture or not?

Lee *et al.*[46] compared the microstructure of two Ti alloy-45S5 Bioglass® composites manufactured by SLM starting from powder mixtures containing either 10 or 30 vol. % of glass. Although the research did not account for complicated geometries, single layers could be successfully fabricated having a controlled circular shape. All composites retained some 45S5 Bioglass® but also contained Ti_5Si_3 derived from titanium alloy and glass reaction. Composites processed from powders with 30 vol. % of glass presented some silicon (Si) in the amorphous phase when a fast scanning speed was applied. To avoid laser-induced reactions between metal and glass, pure metal scaffolds can be printed by SLM and subsequently impregnated with glass. Melo-Fonseca *et al.*,[11] for example, printed Ti–6Al–4V cellular structures and filled them with 45S5 Bioglass® by pressure-assisted impregnation and sintering. As a result of preliminary tests, the heat treatment was optimized at 600 °C to minimize glass devitrification and microstructural changes in the Ti–6Al–4V matrix. This approach is well suited to obtain solid parts for load-bearing implant applications. However, as a drawback, the glass infiltration deeply alters the targeted porous architecture. Moreover, the postprocessing pressure-assisted impregnation and sintering steps substantially increase the manufacturing time and cost.

AM of Ti-bioactive glass composites is clearly a demanding task. On the other hand, there are very few examples in the literature of successful fabrication of Ti-bioactive glass composite parts even by conventional manufacturing methods. Further, none of them offers the unrivalled freedom of geometry that is peculiar to AM. Jurczyk *et al.*[47] synthesized nanostructured-type Ti23Mo-x wt. % 45S5 Bioglass® (x = 0, 3, and 10) composites by mechanical alloying and powder metallurgy methods. The bulk samples were anodically etched to modify their surface topography. Macropores were formed where previous defects had been created upon sintering, whereas mesopores were formed by grain-boundary etching. A ceramic Ca–P layer was cathodically deposited to improve bone tissue integration. In this study, the controlled sintering step at 800 °C led to the formation of a nanostructured Ti23Mo alloy with a grain size of approximately 40 nm.[47] The β phase was predominant in the metal matrix, but the presence of α phase increased with increasing glass weight fractions. The sintering step did not

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TABLE I. Fabrication of titanium-bioglass® composites via (A) additive manufacturing (SLM, Ref. 11) and (B) traditional manufacturing (mechanical alloying, Ref. 47).

	Additive manufacturing	Traditional manufacturing
Composite	Ti6Al4 V SLM impregnated with bioglass (2.05–2.50 wt. %)	Nanostructured β type Ti23Mo-x wt. % 45S5 Bioglass (x = 0, 3, and 10) composites
Printing technique	• Ti6Al4 V produced by SLM • Sandblasted for 30 s • Bioglass pressure-assisted impregnation and sintering for 2 h	• Mechanical alloying lasted for 30 h for all cases • Ball milling was stopped every 2 h • Powders uniaxially pressed at a pressure of 500 MPa • Compacts were heated for 1 h and 30 m • Electrochemical etching
Outcome	Single layers were fabricated having a controlled circular shape	Nanostructured Ti23Mo alloy with a grain size of approximately 40 nm

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induce relevant reactions between metal and glass. Although it was proved that the electrochemically biofunctionalized (etched and Ca–P coated) surface supported osteoblast cell growth and proliferation, this process required substantially longer fabrication times compared to AM (Table I) and produced very basic geometries.

IV. NOT A CONCLUSION, BUT AN OUTLOOK

AM of new composite materials requires further understanding of their printability. For powder-based AM techniques, the properties of the feedstock particles govern the processing efficiency as well as the microstructure and properties of the printed parts. When we look at new composites such as Ti-Bioglass, the melt pool dynamics including total energy, thermal exchange, and melting behavior of different materials are important. Materials that exhibit huge variance in thermophysical properties require a careful adjustment of the processing parameters to avoid printing failure. Properties such as density, laser absorbance, molten viscosity, thermal conductivity, and heat capacity are substantially dissimilar for metals and glasses. The successful production and, later on, the structural integrity in service conditions of composite parts fabricated by AM depend on the qualitative and quantitative agreement of the physical and mechanical properties of the constituent phases, including coefficient of thermal expansion, deformation behavior under load, and fatigue strength.

3D printing a composite material that includes 45S5 Bioglass® is particularly problematic. 45S5 Bioglass® devitrifies before sintering in a temperature range between 550 and 850 °C. The crystallization may improve the mechanical properties of the printed part but it may also impair the original bioactivity of the glass, since it has been reported that the newly formed crystal phases are scarcely bioactive. Moreover, the chemical behavior and hence the bioactivity of 45S5 Bioglass® are dictated by its composition, especially by the proportion of its constituent oxides. Since the glass composition may change because of processing-induced reactions with the metal matrix or because of unexpected interactions with the laser beam, the exact composition of the glass after printing should be carefully checked to estimate the degradation rate postimplantation.

Since geometric freedom is the key advantage of AM, a point of discussion is the feasibility of complicated shapes when composite powders are processed. Whereas the printing accuracy by SLM and by LMD has been clearly demonstrated for Ti and Ti alloys, very little is known about the printing accuracy of composite powders.

Powder storage before printing and recycling conditions after printing represent an additional constraint. It is well known that inappropriate storage of fine metal powders can result in metal oxidation and health problems for users. Also, bioactive glasses are sensitive to moisture and, therefore, water uptake from the environment must be strictly avoided during powder storage to avoid particle agglomeration and to preserve the bioactivity of the glass. Optimal postprinting conditions for powder recycling and ultimately for powder disposal are significantly shaping the development of new materials.

On a broader note, metal-based AM is a young technology. Substantial research in this field remains devoted to the technological improvement in printers, rather than to the development of new feedstocks. Understandably, the palette of metal powders for AM that are currently offered on the market is substantially limited with respect to conventional technologies. However, the revolutionary idea of printing multimaterial systems is opening unexplored scenarios, where geometric freedom and materials functionality grow side-by-side. Now, an additional effort is required to shift from "materials processing" to "multimaterials additive manufacturing" to create a synergistic correspondence between technological progress and materials advancement.

DATA AVAILABILITY

Data sharing is not applicable to this article as no new data were created or analyzed in this study.

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