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Open Celled Porous Titanium**

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Among the porous metals, those made of titanium attract particular attention due to the interesting properties of this element. This review examines the state of research understanding and technological development of these materials, in terms of processing capability, resultant structure and properties and the most advanced applications under development. The impact of the rise of additive manufacturing techniques on these materials is discussed, along with the likely future directions required for these materials to find practical applications on a large scale.

1. Introduction

Porous metals are tremendously varied materials, which attract interest due to their ability to display certain properties with are not inherent to the materials they are made from; the dependence of properties on both the base metal and the structure also means that different properties can be adjusted within certain limits to meet the requirements of applications^[1]. Porous metals have been reviewed in detail on a number of occasions, for example ^[2, 3], but in this review, we concentrate on structures made in titanium in particular. This is because the advantageous mechanical and chemical properties of titanium help to make these materials particularly attractive for a number of applications. While it is possible to process titanium with closed porosity (i.e. pores isolated from each other, not connected to

the outside environment) ^[2], the majority of methods result in open cell structures, sponges, or more recently with developments in Additive Manufacturing (AM) methods, regular structured lattices. This review considers all of these routes, with the main focus given to the methods for random porous materials, as AM for all structures, not just porous, is well described elsewhere ^[4, 5]. Nevertheless, the capabilities of this processing technique with respect to the creation of lattices are important for the future of the method, as well as for future fabrication of porous titanium ^[6].

2. Processing & Structure

It is well known that processing titanium in the liquid state can be very challenging due to its high melting temperature and its extreme affinity for atmospheric gases such as oxygen, carbon and nitrogen ^[2]. Therefore, Ti foams are almost always processed by powder metallurgical routes, generating and retaining a porous form from powder particles, and causing atomic level bonding between the particles with a sintering treatment at a temperature below the melting point. It is also possible to fabricate alloys, such as the shape memory NiTi alloy, by the alloying of elemental powders during the sintering step ^[7, 8]. Focusing on powder based production methods used for titanium and its major alloys, it is still possible to delineate several different types of process, which are summarized in **Fig. 1**, and discussed in the following sections. Note that Fig. 1 indicates how the AM methods stand somewhat apart from the other techniques in this regard.

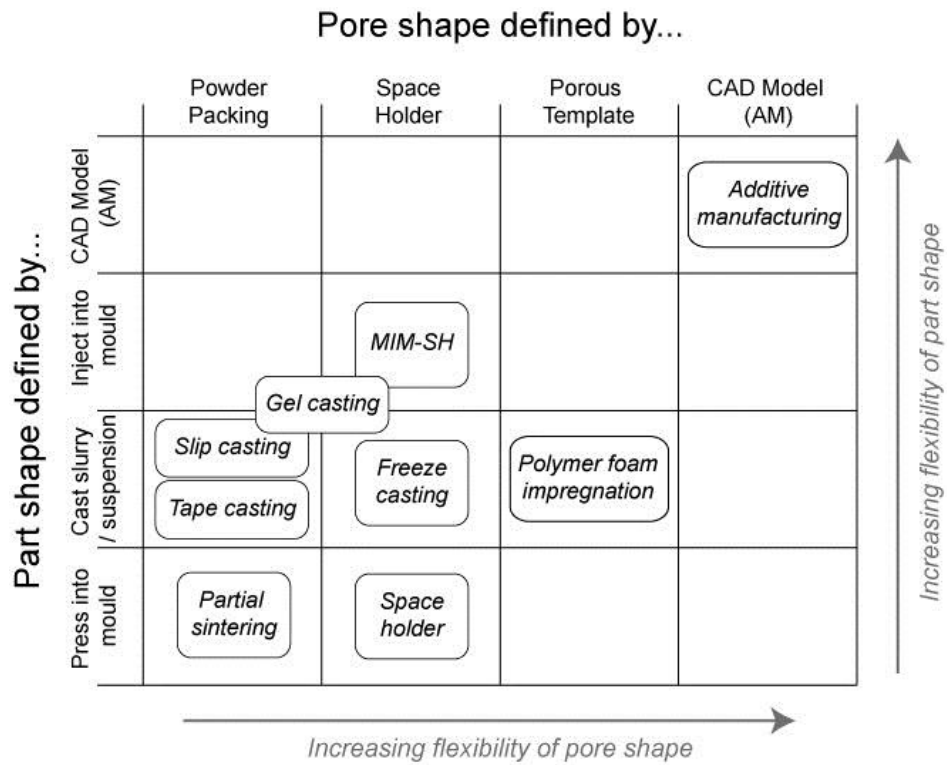


Fig. 1 – A schematic diagram classifying many of the processes reported for processing of open cell porous titanium. Because of their preponderance in the literature, only powder metallurgical methods are included. All methods are discussed in more detail later in the section.

2.1 Partial Sintering

One of the simplest ways of producing porous Ti is to partially sinter the metal powder under vacuum (**Fig 2**). This will result in the initiation of small necks among the particles via diffusion and the retention of relatively small pores due to incomplete densification, provided the temperature and duration of the sintering step is appropriately controlled (sufficient to cause inter-particle bonding, but not enough to reach near full density). The process has been used for the manufacture of Ti with a total porosity of up to 60 %, showing an elastic modulus of 5-60 GPa^[9]. The sintering characteristics, and hence porous structure, are affected by the size of the starting powder. Large particle sizes have greater inter-particle spaces, and will result in larger pores, along with increased porosity due to a reduced rate of sintering, with the opposite true for smaller particles^[10]. Pore shape of course cannot be controlled and careful control of the sintering procedure is critical; many foams that are processed by sintering as

part of another method will contain some degree of microporosity even within the nominally dense metal parts, which can have a significant impact on overall porosity and on properties.

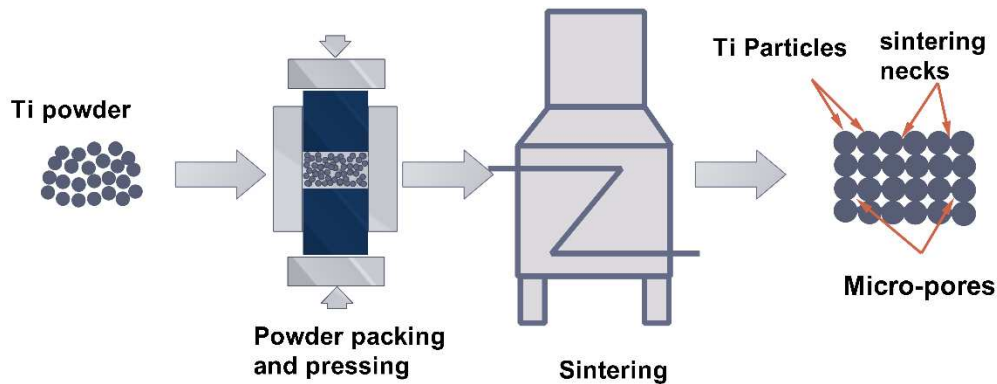


Fig. 2 – A schematic diagram of the partial sintering technique.

2.2 Slurry-Based Methods

2.2.1 Slip Casting

Slip casting is a method commonly used in the ceramic industry, but more recently applied to porous Ti [11, 12]. A suspension or slip is first prepared by mixing Ti powder with a solvent and dispersant such as water and cyclohexane. A stabiliser and polymeric binder are usually added to the mixture to increase the stability of the solid part in the suspension and provide sufficient strength for later handling. This suspension is then poured into a porous gypsum or plaster mould which absorbs the water from the suspension by capillary action, resulting in a green body with a very limited strength^[13]. This cast body is then removed from the mould and dried for a period ranging from 8 to more than 24 hours before sintering. This method was successfully used for the production of Ti foams with a total porosity of up to 65 %^[12], slightly higher than that achieved for partial sintering of powders, possibly as the mechanical compaction of the powders pre-sintering is less. Like partial sintering, the control of the pore size is low, and inhomogeneous structures may result. One of the drawbacks of the technique from a processing point of view is the long drying stage, particularly when large parts are produced, leading to greater suitability for small scale production^[13], however the capital cost is low with relatively

simple tools ^[11]. Another potential issue is contamination of the foam during thermal removal of the stabiliser and binder (which are usually polymers).

2.2.2 Tape Casting

This process also uses a slurry of Ti powder with a solvent, binder and disperser. A small amount of TiH_2 may also be added to the mixture to facilitate the sintering process by releasing hydrogen into the sintering atmosphere as it breaks down at high temperature, reducing oxidation. The homogenised slurry is then poured into a chamber situated above a moving carrier film or foil, **Fig 3**. The slurry passes through a small gap controlled by a doctor blade and is then left to dry at ambient temperature or in an oven to remove the solvent. After that the sheet produced is thermally debound to remove the organic binder and is then partially densified by sintering. This method has been used for producing porous Ti foil with a thickness of $370\ \mu m$ and a total porosity of 36 %, with once again pore sizes limited to micropores ($22\ \mu m$ average pore size in this case) ^[14]. These porous sheets could have potential in several applications, such as in chemical reactors and fuel cells ^[14].

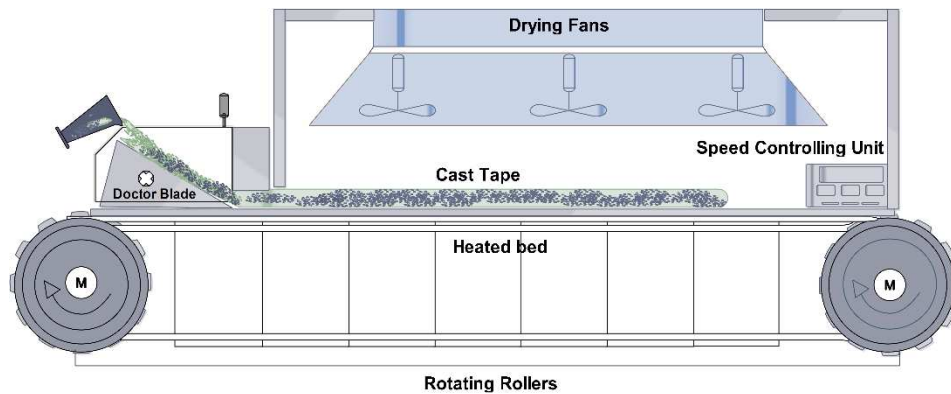


Fig. 3 – A schematic diagram of the tape casting process.

2.2.3 Gel Casting

In this process, **Fig 4**, the slurry is prepared by mixing the Ti powder with a monomer and cross linker, to form a gel, and deionised water, to dilute the mixture. A small amount of a disperser is added to the slurry to prevent Ti segregation and settling. After that an initiator and catalyst are added to start the polymerization reaction, which forms a gel; this is agitated, before being cast into a mould and heated at a temperature of 60 °C for 2 hours^[15]. Next, the gelled parts are demoulded and vacuum dried at ambient temperature for 24 hours before sintering under vacuum to produce a porous Ti part. As the internal structure of the gel plays a role in defining the porosity (although the degree of control thus achieved is not great), this process has the elements of a space holder method, where another phase defines the shape of the pores. A total porosity of 46 % has been achieved^[15], which is relatively low in comparison with other methods and attempts to increase this have been made^[16] by first producing a gelled TiO₂ foam which was then reduced to metallic Ti by electrochemical reduction, reaching a total porosity of 88 %. However, the linear shrinkage observed on the reduction stage was substantial (27%), and a TiC phase is formed in the material which has a detrimental effect on the ductility.

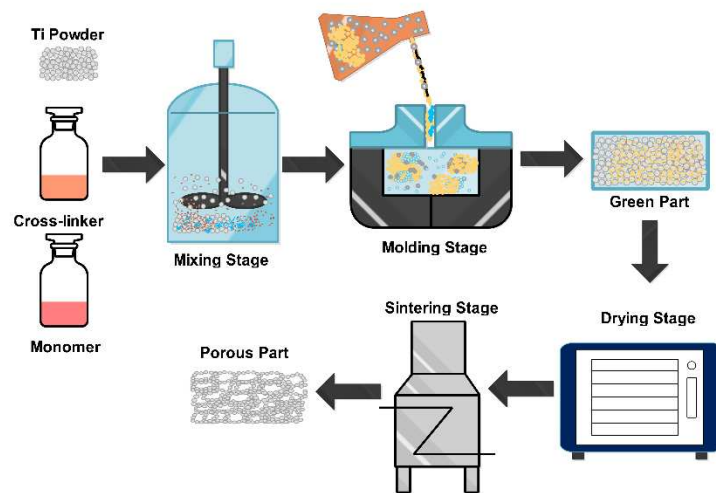


Fig. 4 – A schematic diagram of the gel casting process.

2.2.4 Freeze Casting

The freeze casting process was first used for the manufacture of polymer scaffolds for biomedical applications (it is well known, as freeze gelation, in the polymer industry ^[17]) and has been used for the production of ceramic biomaterial scaffolds ^[18]. It was implemented for the production of porous Ti in 2008 ^[19, 20]. Like other methods, the first step is the preparation of a slurry or suspension, with water the most commonly used solvent (though this presents a risk of oxidation). This suspension is then unidirectionally solidified by freezing, **Fig. 5**, leading to the formation of dendritic ice crystals among the metal powder particles. These growing ice crystals reject and push the Ti particles to the boundaries or spaces among the crystals, concentrating them in these regions and thus creating lamellar cell walls in between. The dendritic ice crystals are subsequently sublimated by freeze drying under vacuum, forming dendritic pores that replicate the shape of the crystals; the ice thus acts as a removable space holder phase. Finally, the porous cast is thermally debound and partially densified by sintering. Due to the directional solidification, the macropores are often elongated and aligned along the solidification direction, with a high aspect ratio. Indeed, the pores frequently form channels traversing one direction across a sample without interconnects in other directions, and therefore are potentially well suited for filter applications. The critical role of water solidification means it is vital to control the cooling rate, which affects crystal formation, and the particle size of the powder, which affects its mobility and densification behaviour (this is particularly significant as, compared to other materials processed by freeze casting Ti has a high density and tendency to low mobility and degradation). Very fast cooling rates can result in the formation of dense material ^[21], while large particle sizes can result in particle entrapment by the crystals giving lower porosity (for example 38 %^[19]) and smaller, non-elongated pores. A total porosity of up to 69 % has so far been achieved by this method ^[22].

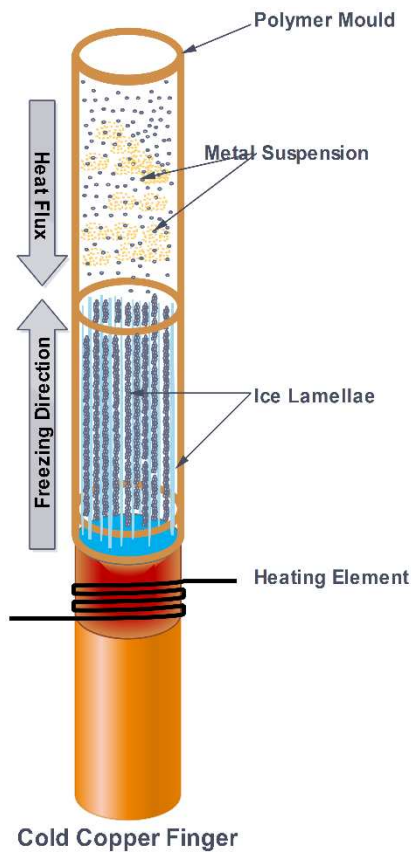


Fig. 5 – A schematic diagram of the freeze casting process.

2.2.5 Impregnation of Polymer Foam

This process, **Fig. 6**, also frequently involves a slurry, but rather than being shaped or cast into a mould, polyurethane foam is dipped into it, with the slurry impregnating the foam. Excess slurry is removed by squeezing or pressing it under a roller ^[23], and the foam is left to dry before thermal treatment to remove the binder, and also the polymer foam by pyrolysis. This method can yield Ti with high porosities (up to 84% ^[24, 25]) and good, well-controlled structures, leading to efficient mechanical properties (albeit with the ever-present risk of contamination due to binder removal).

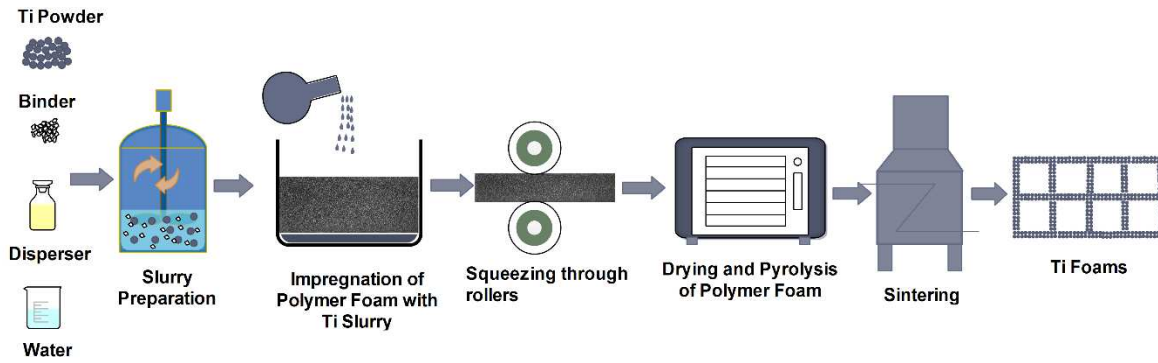


Fig. 6 – A schematic diagram of the impregnation of a polymer foam to produce porous Ti.

2.3 Space Holder

The degree of control over pore size and shape, and to a lesser extent, porosity is limited with the methods discussed above where the pores develop during the process. In order to increase control, and achieve the highest porosities, a simple and widely-used strategy is to incorporate particles of a phase within the material which does not react or change significantly during the process, and can be removed at some stage, leaving behind open (and interconnected) pores. Such a material is known as a *Space Holder* (or sometimes *porogen*).

The space holder needs to be low cost, easily removed and readily available with the desired shapes and sizes. Furthermore, it must withstand any applied pressure during processing without deforming. In addition, it must not react with the Ti to avoid undesirable phases that can have detrimental impact on the mechanical properties of the foam. NaCl is frequently used as a space holder (with a full list of space holder materials used for Ti given in **Table 1**). Although NaCl will undergo sintering^[26], the goal in the process is simply to retain the physical space occupied (though a degree of interconnection between the particles will aid space holder removal). In liquid state processed foams, such as aluminium, the space holder can be tailored to complex shapes^[27] but for powder methods where the metal and space holder are combined together space holders are normally used in the as-received powder condition.

Table 1 – Space holder materials used for the production of porous Ti.

Material	Method	Removal	Comments	Ref.
Carbamide (Urea)	Space holder + Cold Isostatic Pressing	Thermal Removal [28] 200 °C for 2 hrs [29] 300 °C for 2 hrs. [30] 200 °C for 3 hrs+350 °C for 3 hrs	Low melting point (133°C). Very good water solubility. Possibility of deforming. Breaking at pressure > 200 MPa [31]	[28-30]
Ammonium Bicarbonate	Cold Isostatic pressing	Thermal Removal [32] 120 °C for 3 hrs [33] 95 °C for 12 hrs	Very low melting point (41.9 °C). Unsuitable for MIM and HIP	[32, 33]
Sodium Chloride	Space holder	Water Dissolution [34] 4 hrs at 50-60 °C. [35] 60 °C for 5 hrs	High melting temperature (801°C). Good water solubility & low cost	[34-38]
	MIM+Space holder	[35] 60 °C for more than 72 hrs [39] 50 °C for 40 hrs (92% removed)		
	Warm compaction Hot Pressing	[36] 24 hrs (96% removed) [38] 2-3 hrs		
Potassium Chloride	Space holder	Water Dissolution 60 °C for 5 hrs	Good water solubility & low cost. Available in different shapes and sizes	[35, 40]
	MIM+Space holder	[35] 60 °C for more than 72 hrs		
	Warm compaction	[40] 60 °C for 24 hrs		
Starch	Space holder	Thermal Removal 450 °C for 2 hrs	Low cost, high contamination	[41]
Saccharose crystals	Space holder	Water dissolution with magnetic stirrer at 20-80 °C for 2-6 hrs	Very good solubility in water & low cost. Very hard to use in MIM [42]	[43]
Poly(methyl methacrylate)	Space holder	Thermal Removal [44] 200-450 °C for 2 hrs.	Can be used as a binder and as a space holder	[39, 44]
	MIM+Space holder	Solvent Dissolution [39] Acetone 40 hrs (90-100% removed)		
Polyoxymethylene	Space holder	Catalytic decomposition at 110 °C	Melting point 175 °C.	[45]
Polypropylene carbonate	Space holder	Thermal Removal 220 °C for 1 h	High carbon contamination	[46]
Acrowax	Space holder	Thermal Removal 200 °C for 3 hrs.		[47]
Hydrogen Carbonate	Space holder	Thermal Removal 200 °C for 5 hrs.		[48]
Magnesium	Space holder	Evaporation by heating	Relatively expensive. Presence of MgO [49]	[50]
Steel spheres, wires and mesh	Space holder	Electrochemical Dissolution 10-20 hrs	Expensive. Hard to remove completely	[51]

The aim is that the space holder imparts and defines the pore size and shape, yet there are several processes at work during or after space holder removal in many processing methods, and this relationship may not be exact. **Table 2** summarises the relationships between quoted space holder size ranges and those of pores. While commonly the same or very similar, smaller distributions of pores have been reported ^[52, 53]. The assessment could be complicated by space holder deformation during compaction, which has been noted to occur and result in pores with elliptical shape ^[54].

Table 2 – The relationship between space holder size and pore size reported for porous titanium

Space holder	Size of space holder (µm)	Size of final pores (µm)	Process	Pressure MPa	Sintering temp. °C	Time (h)	Ref.
Sodium Chloride	50-500	50-500	SH	30-50	Hot pressing 780 °C	2	[55]
Sodium Chloride	< 290	50-300	MIM+SH	100	1150 °C	2	[56]
Sodium Chloride	300-500	50-500	MIM+SH	-	1000 °C 1200 °C	4	[57]
PMMA	D ₅₀ =600	50-500	MIM+SH	-			[57]
Table sugar	800-1000	800-1000	SH	500	1250 °C	1	[43]
Ammonium Hydrogen Carbonate	200-500	200-500	SH	100	1200 °C	2	[48]
Carbamide	800-2400	100-2500	SH	166	1400 °C	1	[58]
Ammonium hydrogen carbonate	100-900	Smaller	SH	166	1400 °C	1	[58]
Ammonium hydrogen carbonate	500-800	300-800	SH	-	1200 °C	10	[53]
Polypropylene carbonate	100-600	50-500	SH	100 400	1000 °C	2.5, 5 or 10	[59]
Starch	100-400	100-300	SH	100	1200 °C	3	[41]
Polyoxymethylene	D _{mean} = 500	80-400	SH	50-100	1300 °C	2	[60]
Acrowax	300-500	300-500	SH	100	1100 °C	2	[47]
Potassium chloride	188-476	100-480	MIM+SH	45	1250 °C	2	[61]

2.3.1 Pressing with Space Holder

To create actual porous metal, the space holder needs to be combined with the powder and shaped. Simple pressing (hot or cold) of a blend of metal powder and space holder is one of the most widely reported methods. After this stage the space holder will be removed and the metal sintered to allow further integration of the powder. A schematic of the method is shown in **Fig 7**.

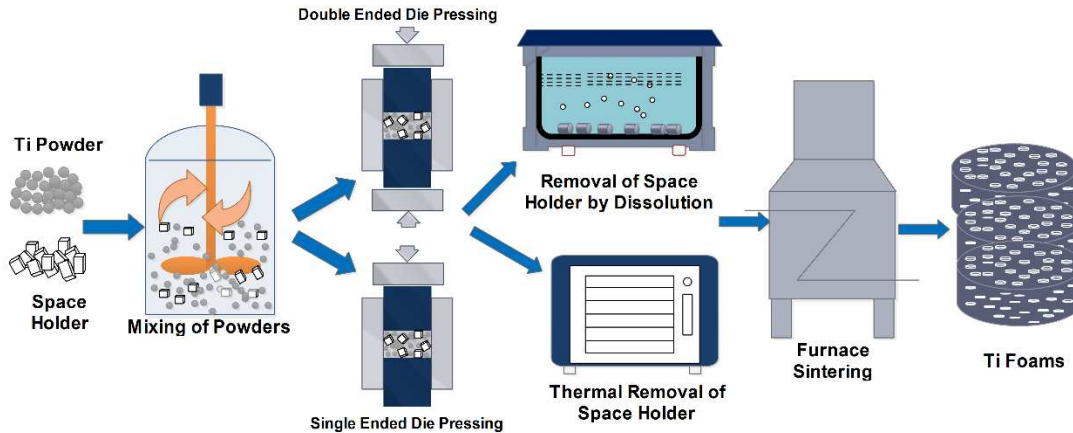


Fig. 7 – A schematic diagram of the space holder technique.

Several techniques (all taken from the powder metallurgy of conventional materials) have been used for the compaction of the Ti powder-space holder mixtures such as single and double ended cold axial pressing [41, 43, 52, 58, 60, 62, 63], Uniaxial Hot pressing (HP) [55] and warm compaction [35, 64, 65], producing titanium with a total porosity of up to 80 % [58, 66]. As well as the typically limited range of part geometries that can be created, a drawback of applying these techniques is that the pressures involved are high, and this can cause the space holder particles to deform and break during compaction, altering the final shape of the pores. For example, it has been reported that NaCl space holders are broken at a pressure higher than 350 MPa [34], and it has been noted that the space holder particles are deformed and flattened in the direction of the applied pressure during HP, leading to flattened pores with deformed shapes [55].

Where single ended uniaxial pressing is used, the pressure profile is non-uniform throughout the compacted specimen (higher at the top and in the centre ^[67]) thus leading to non-uniform mechanical properties; correlations have been found with the expected pressure distribution and the fracture lines in tested samples ^[67]. As metal powder and space holder sizes and densities are frequently different, segregation can occur, which results in inhomogeneous distribution of the space holder particles in the Ti powder and a reduction in pore interconnectivity; to overcome this a polymeric binder may be included in the mixture^[60, 67, 68].

Normally, the compacted blend of these powders is sintered in a furnace under vacuum or flow of an inert gas. However, there have been attempts to use faster and more advanced sintering techniques such as pressureless Spark Plasma Sintering (SPS) ^[69] as well as conventional SPS under uniaxial pressure ^[70].

2.3.2 Metal Injection Moulding with Space Holder (MIM-SH)

Injection moulding is a net shape manufacturing process originally designed for mass production of plastic parts, which has been adopted for use with metals. Combining this with the use of space holders could have huge potential in mass producing porous Ti with near net complex shapes, overcoming some of the limitations of the space holder methods on their own ^[35], especially when the potential for pore structures to be closed by debris or altered by deformation during machining is considered ^[71].

There are four key stages to the process, **Fig. 8**; first a feedstock is prepared by mixing Ti powder with a polymeric binder and space holder, which is then granulated by extruding and cutting into small pellets; secondly the feedstock is injected at a relatively low temperature and pressure into a die cavity which imparts the desired shape; thirdly the space holder and binder are removed by different techniques (thermally or with a solvent) depending on their nature; finally, the porous green part is partially densified by sintering ^[61, 72]. Although relatively little employed in comparison to the other space holder methods (due to the charge volume required for most injection moulding systems

precluding small scale research production), the method has been used to produce commercially pure Ti with a total porosity of up to 71% [56]. Examples of the materials produced both macroscopically and microscopically are shown in **Fig. 9**.

To overcome the difficulty of full size experiments, the process is sometimes simulated by warm compaction, heating and pressing the feedstock at a temperature and pressure equivalent to the temperature and pressure used in MIM [35, 36, 65], however, without the flow of feedstock into the mould the accuracy of such simulations to actual MIM processed parts is poor; for example, feedstock with a solid content of 80% was successfully warm compacted, but clogged the nozzle during MIM, due to powders segregating on injection [35].

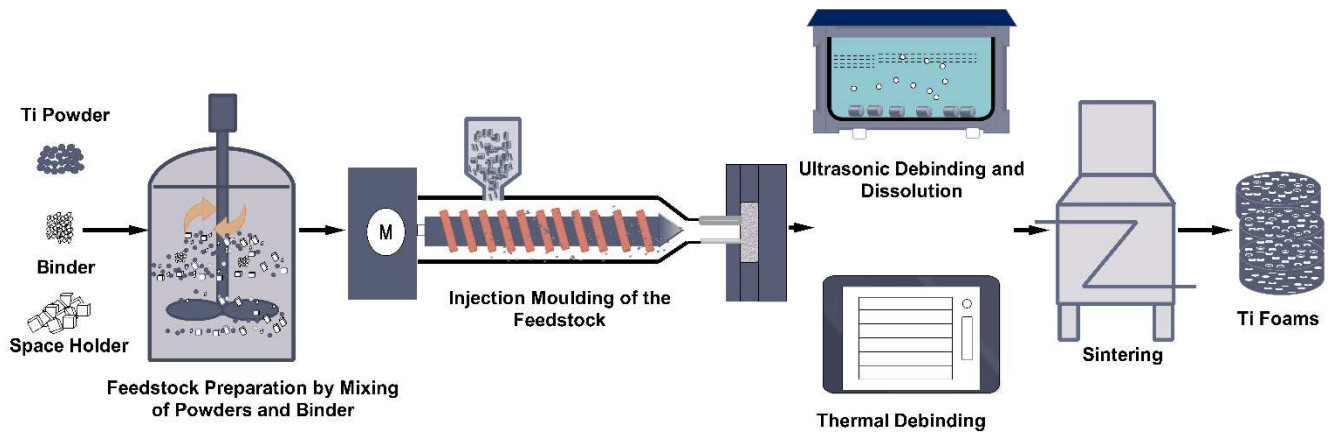


Fig. 8 – A schematic diagram of the MIM-SH technique.

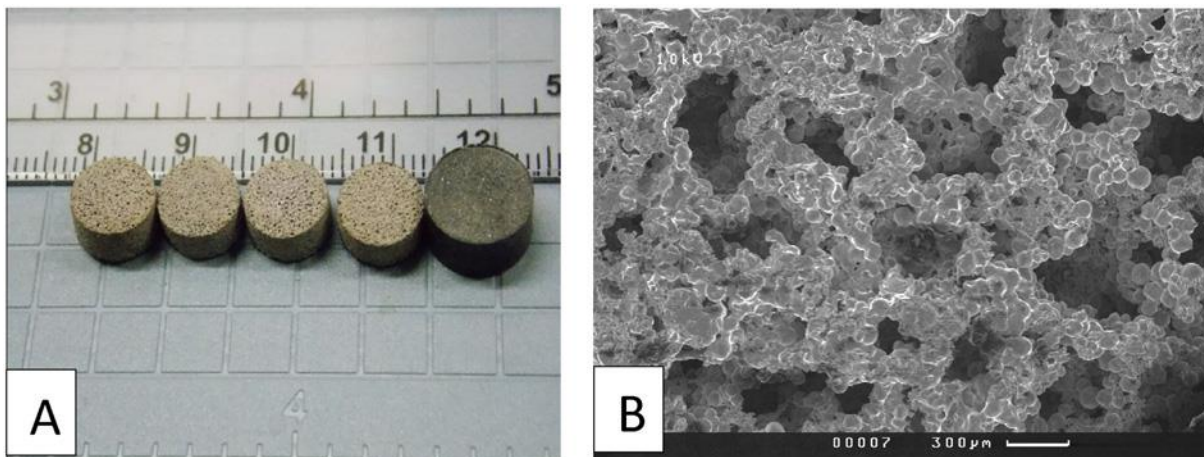


Fig. 9 – Porous Ti processed by MIM-SH. a) four sintered and one green state sample, and b) an SEM image of a sample post sintering. Reprinted from *Materials & Design*, 87, MM Shbeh, R Goodall, Design of water debinding and dissolution stages of metal injection moulded porous Ti foam production, 295-302, Copyright (2015), with permission from Elsevier

2.4 Additive Manufacturing

Additive Manufacturing (AM) techniques begin with powder and use a directable heat source (such as a laser or electron beam) to melt certain volumes, according to a 3D model specified in a computer. Shapes are frequently built up layer by layer, which may involve sequential deposition of powder layers, with the final part being removed from the unmelted powder on completion, **Fig. 10**. The geometrical freedom of such methods means that they are highly suitable for the production of porous materials, especially regular structured lattices, although the requirement to remove powder from free volume means they are restricted to structures with a high degree of openness (as most lattices of low relative density are).

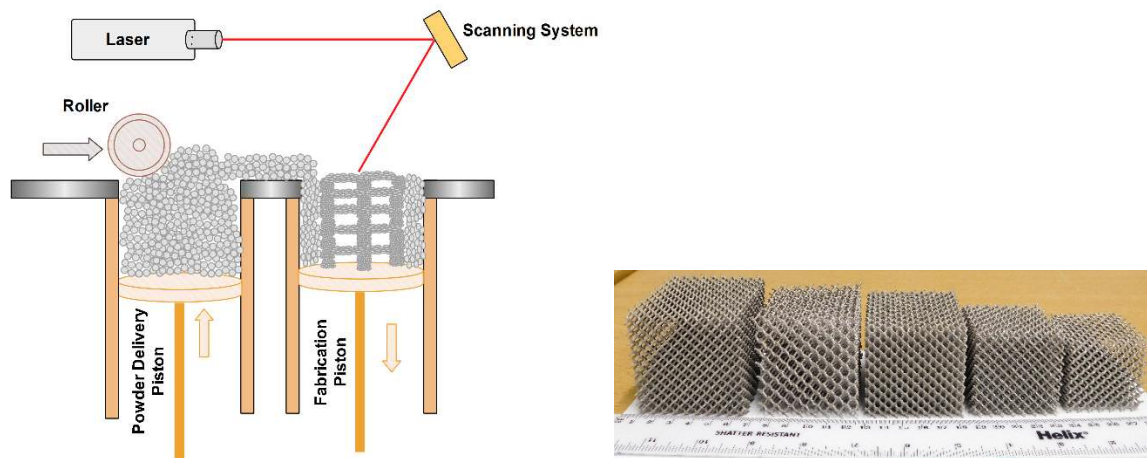


Fig. 10 – A schematic diagram of a generic Additive Manufacturing process. A layer process using a laser, such as *Selective Laser Melting*, is depicted, although process specifics will differ with method and device. Also shown is an image of various titanium lattices produced by *Electron Beam Melting*.

Additive methods have recently been widely used to fabricate regular lattice structures from titanium, and even random structured porous sponges (produced by using X-ray Tomography to generate an image of a random pack of glass spheres, and then inverting the image to produce the build file ^[73]). As an example of the typical use, Selective Laser Melting (SLM) has been used to make Ti lattices with octahedral unit cells of relatively large size (600-1400 μm) over a wide range of porosities (reportedly 10 to 95 %), achieved by varying the strut thickness via the laser energy and spot size ^[74]. Ti lattices with more complex shapes such as square pyramid, truncated cube, diamond, truncated cuboctahedron, rhombic dodecahedron and rhombicuboctahedron have also been recently produced by this technique ^[75, 76]. One challenge is finding the optimum building parameters for each metal powder in order to produce lattices with as low a defect (e.g. partially fused particles or porosity) content as possible. Porosity reduces mechanical properties and adhered particles at the surface which may be liberated are a concern for biomedical applications. Some studies have suggested the use of post manufacturing heat treatment in order to fuse these un-melted particles and enhance the strength of the Ti lattices ^[76, 77], while others investigated the use of jet blasting followed by sintering in order to remove the partially fused particles, finding this is an effective technique, though it can result in fracture and bending of struts, especially near the surface ^[78].

An alternative technique is Electron Beam Melting (EBM), sometimes preferred due to faster builds as the electron beam can be deflected electromagnetically, not requiring the motion of physical optics. The method has been used for the production of Ti lattices of various designs including those based on unit cells with structures of diamond ^[79], cubes ^[79, 80] and more complex cubic-based designs ^[79] as well as rhombic dodecahedron ^[81].

Lattice structures not based on conventional unit cells, such as tensegrity structures, have also been produced by EBM and experimentally used to validate predictions of mechanical response ^[82, 83].

Lattices naturally have properties that depend on the base material and its spatial arrangement (linked to such characteristics as the porosity), and Additively Manufactured materials can have significant effects from processing conditions (e.g.^[84]), but for AM lattices there is also significant interaction between the designed structure, the fabrication process and the properties obtained, resulting from the effect of the shapes built and the population of defects, and the microstructure^[80]. Measured properties of lattices are discussed in section 3.1.

2.5 Non-Powder Based Methods

Other processing methods do exist, and can have particular advantages. For example, those based on existing Ti processing technology may have the advantage of more easily being brought to industrial scale. An example of this is the use of investment casting with rapid prototyping methods, such as multijet modelling^[85], used to produce a complex porous structured wax or polymer pattern to a CAD design which is used as the investment^[86]. This method has been used to produce Ti with a total porosity of 60 % and a compressive strength of 80 MPa, though it should be noted that the surface contamination of carbon was very significant (being measured by XPS at 62.7 at%) which is unlikely to be optimal for titanium or its alloys. It is also possible to use the same process to form a wax space holder, which is then filled and pressed with a powder-containing slurry and pressed under heat to form a Ti-wax composite. The wax is removed by dissolution in xylene and the Ti is then sintered, the process having been used to produce Ti with a porosity of 66.8 ± 3.6 % and an elastic modulus of 20.5 ± 2 GPa^[87].

3. Properties

The properties of porous materials have been widely examined, and a range of behaviours found. Certain properties, such as the electrical conductivity^[88] depend on the base metal behaviour in a

relatively simple way (provided full consolidation within the struts is reached, a key aspect of powder processed porous metals), and can be normalized to give consistent foam behaviour relative to the dense material, depending on key structural parameters such as the porosity. Other properties, such as mechanical behaviour, have more complex dependence on the material condition and microstructure as influenced by the processing required to generate the porous structure, and will be discussed in more detail here.

3.1 Mechanical Properties

3.1.1 Uniaxial

By far the most frequently applied test for the mechanical response of porous materials is compression. As a result, there is a significant amount of data available for the uniaxial response of various porous titanium samples (mostly compression, though some instances of other tests have been reported, e.g.^[89]). These data, for the most commonly reported processing methods and materials are presented here in a series of graphs (full data collated from the literature may be found in the Supplementary Information). The elastic response is characterised by the Young's modulus, E . Data from the literature is collated in **Fig. 11**, showing how the value of E changes with the relative density of a series of randomly-structured porous samples made from non-alloyed titanium. The majority of reported data are for foams processed by the space holder method (albeit using a variety of space holders), MIM-SH, freeze casting and gel casting, and a small number of points for the impregnation of polymer foams. A number of lines showing the predictions for simple models for the elastic behaviour of a foam (the Rule of Mixtures upper limit and the Gibson Ashby model for 4 different values of the constant C , from 1-0.1) are given. For the plot, variations in processing conditions and pore sizes have been ignored in classifying the data into different series (see Supplementary Information for details). This may be one of the reasons behind the large variability seen within each method, along with minor variations in

testing and analysis procedures. Looking at the data, it is seen that, while there is significant overlap, in the reported data the space holder methods have achieved some of the highest stiffness values (approaching even the upper bound specified by the Rule of Mixtures) at a given density, while MIM-SH is frequently among the lowest. The spread however implies these are more due to variations within the techniques, and not fundamental characteristics. As an additional point, the flexibility of the methods is demonstrated by the large range of relative densities which have been made and tested.

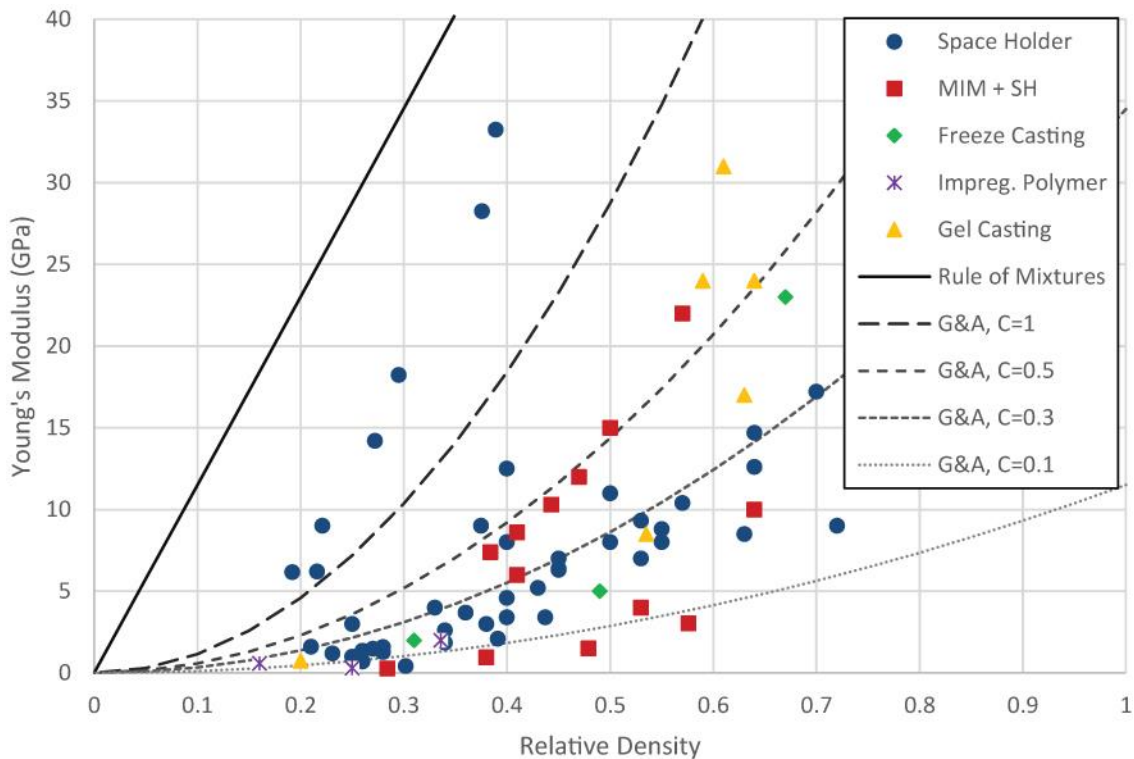


Fig. 11– Reported values for the Young’s modulus of non-alloyed porous titanium with random structures, plotted against the relative density. Data are grouped into the broad class of methods, ignoring variations in how the technique is applied and pore size. Also shown are the prediction lines from the Rule of Mixtures and the Gibson-Ashby approach with different values of the constant, C.

Data are from [15, 20, 22, 25, 35, 41, 47, 55-57, 60, 66, 67, 90-100]

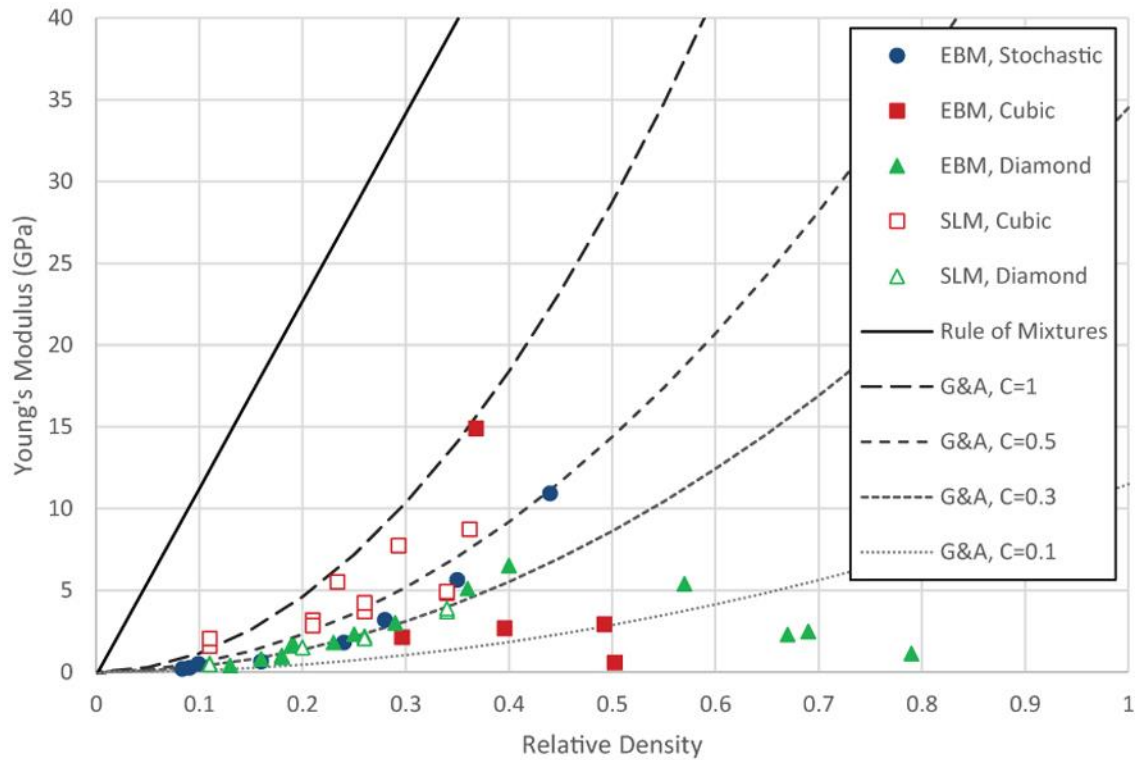


Fig. 12 – Reported values for the Young's modulus of porous *Ti6Al4V* fabricated by AM, against the relative density. Data are grouped by method and structure type, ignoring variations in build parameters and lattice size. Also shown are the prediction lines from the Rule of Mixtures and the Gibson-Ashby approach with different values of the constant, *C*. Data are from ^[73, 101-110].

Fig. 11 shows data (where it has been reported) from all of the methods from Fig. 1 except for Additive Manufacturing. As this technique typically uses *Ti6Al4V* alloy, and there is variation in method and structure design, these data are shown separately, plotted in the same manner, in **Fig. 12**, with further details again available in the Supplementary Information. The values achieved are broadly comparable with porous titanium made in other ways, indicating that the structures included in the plot (those most commonly investigated, random structures replicating sponges, cubic and diamond lattices) are not significantly more mechanically efficient than randomly-produced porosity. The two main processing methods, EBM and SLM, also seem broadly consistent, though variable results are seen for cubic lattices, both high and low for a given density compared to other structures. Low property values at

high relative density may reflect the difficulty in producing a well-defined lattice structure when the amount of free volume is small.

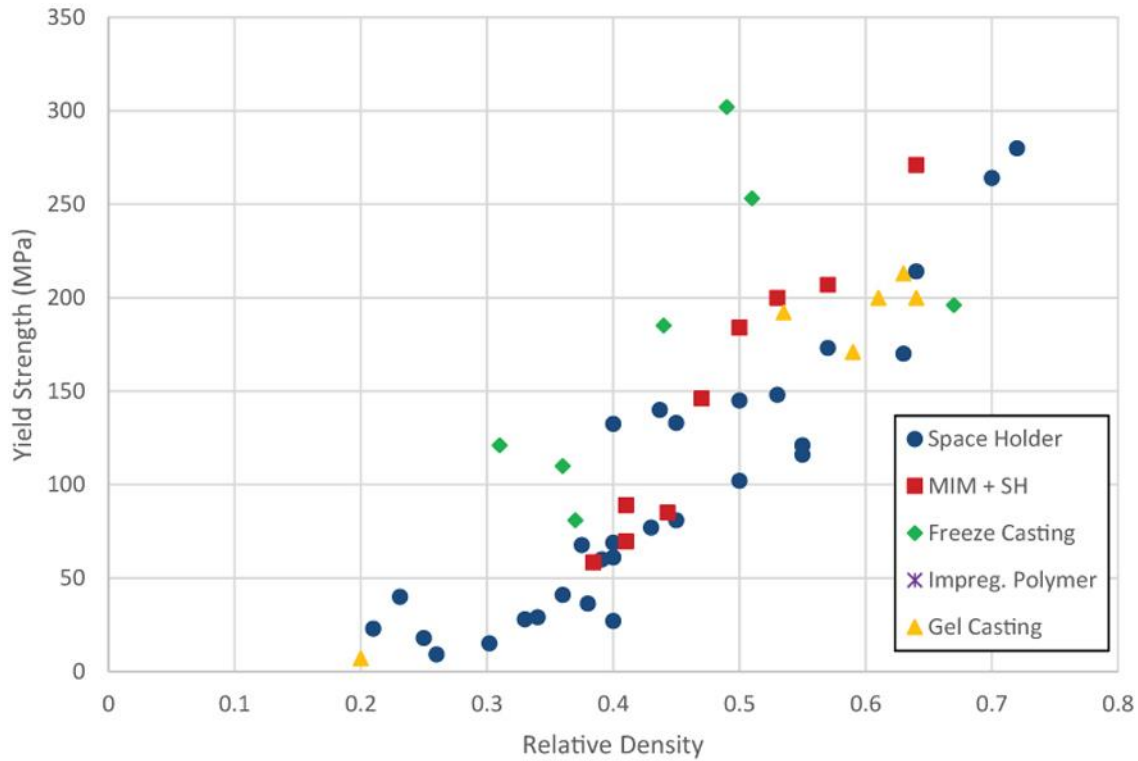


Fig. 13 – Reported values for the yield strength of non-alloyed porous titanium with random structures, plotted against the relative density. Data are grouped into the broad class of methods, ignoring variations in how the technique is applied and pore size. Data are from references given in Fig. 11.

The plastic behaviour of a porous material can be characterized in several ways, leading to a smaller quantity of data which can be analysed together; sometimes the plateau strength is reported, and in other instances it may be the yield (or offset yield) stress. Here, due to the importance of the point of initiation of plastic flow for the applications discussed below and the relatively poor definition of the plateau stress in porous materials that do not show the classical form of the curve with a perfectly flat region, the yield strength is used (taking the offset yield strength as being an acceptable measure where this is reported), and these are collated and plotted in **Figs. 13 and 14** for porous titanium, separating out the Additive Manufacturing methods as previously.

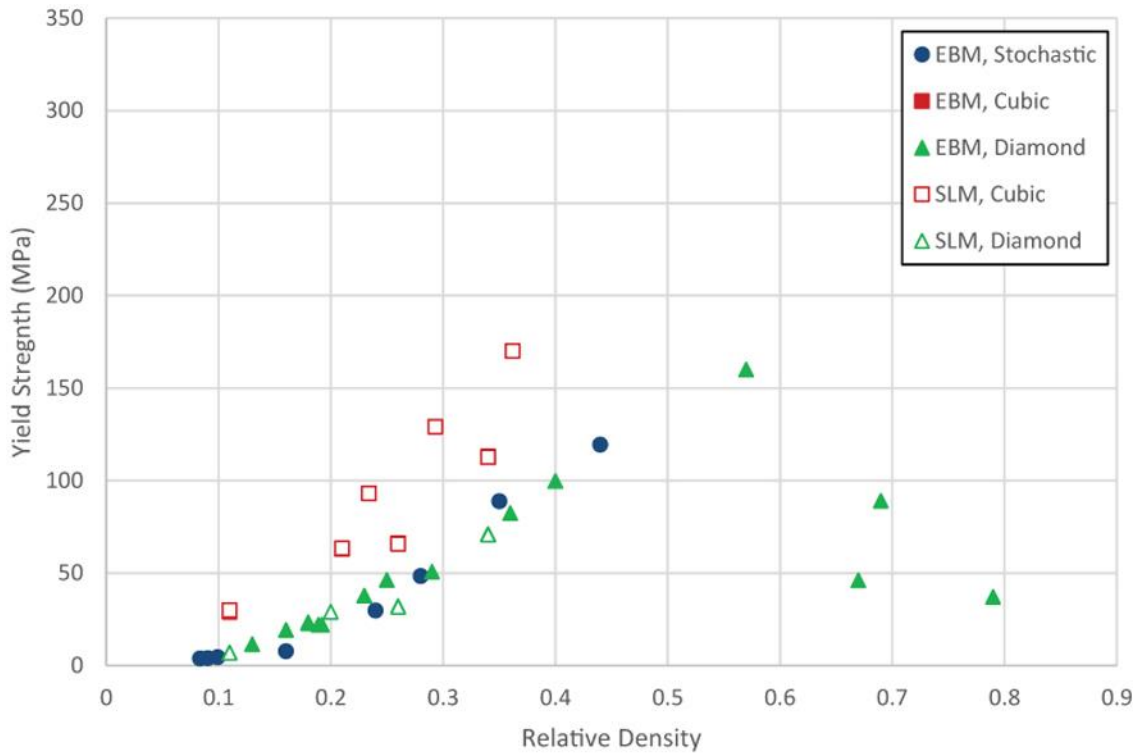


Fig. 14 – Reported values for the yield strength of porous Ti6Al4V fabricated by AM, against the relative density. Data are grouped by method and structure type, ignoring variations in build parameters and lattice size. Data are from references given in Fig. 12.

The strength data in Figs. 13 and 14 show more consistency than the Young’s modulus, without significant differences based on manufacturing method or between the conventional and Additive techniques. This observation is perhaps surprising, as it would be expected that microstructural variation, significant degrees of which would be expected, would affect plastic behaviour more strongly. However, as yield is likely to be affected by defects present (such as misformed pores or missing struts), this may reflect the fact that in such porous materials the structure on the scale of the porosity is determining the transition from purely elastic to elastic and plastic deformation.

3.1.2 Fracture

Fracture behaviour and toughness of porous metals is experimentally more challenging to address, although evidence exists that the behaviour on fracture is more dependent on the deformation mechanisms possible within the dense material ^[111]. There are few studies investigating the fracture toughness of porous Ti in the literature. One example reports the fracture toughness measured in pre-cracked specimens for titanium with 60 and 70% porosity ^[112]. The K_{IC} values were 7.0 and 4.0 $\text{MPa}\sqrt{\text{m}}$ respectively, significantly lower than dense Ti (75 $\text{MPa}\sqrt{\text{m}}$), reduced by a greater proportion than the reduction in material present, in general agreement with proposed scaling laws for the fracture toughness of porous metals ^[113].

3.1.3 Creep

More advanced properties, such as creep, have been experimentally studied for some porous metals including aluminium ^[114, 115], and nickel based foams ^[116, 117] as well as the development of predictive frameworks and analytical models ^[118, 119], but experimental exploration for porous titanium has not been reported, reflecting the recent focus on biomedical rather than high temperature applications.

3.1.4 Fatigue

As porous titanium is likely to be used in applications with some structural role (see section 4), assessing the behaviour under cyclic loading is extremely important. However, the fatigue life of porous materials (particularly those produced by powder methods) is much more difficult to assess than ordinary dense material, due to the presence of macro and micropores, and high surface roughness, including on the internal surfaces. To reduce this dependence, and explore the effect of defects, the fatigue behaviour of Ti with a total porosity of 60% (made by the space holder technique) with a notch machined in it has been explored and compared with another sample having similar percentage of porosity, but coated with a thin layer of solid Ti by localized melting of the surface ^[120]. Samples were

cyclically loaded with a maximum load of 200 N and frequency of 40 Hz. A dominant crack was observed to form in a pre-cracked cell wall and grow continuously along the weakest path until fracture. It was also noted that the uncoated porous Ti displayed a higher Paris exponent ($m=17.1\pm 0.3$) than the coated sample ($m=14.2\pm 0.2$), attributed to the closure and bridging of the cracks ^[120]. Fatigue has also been investigated in porous Ti samples made by the space holder technique ^[121] with porosities ranging from 51-65% under compression-compression fatigue with a frequency of 5Hz and a constant stress ratio of 0.1 up to failure or a million cycles. It was reported that the fatigue limit could be normalized with the yield strength for a particular density, being 0.75 of the normalized maximum stress applied. While not taking samples through to failure, the effect of fatigue-induced damage was tested by applying cyclic compression across stress ranges up to 50-60 MPa (below the compressive yield strength) at a frequency of 6 Hz and a minimum to maximum load ratio of $R=0.1$ in simulated body fluid, to Ti with 62.5% porosity, made by the space holder technique. A reduction in the compressive strength by two thirds was seen ^[33]. Fatigue behaviour of lattices has been explored, similarly finding a reduced fatigue strength, attributed to stress concentration arising at various levels from the surface finish and defects, and the microstructure created ^[122].

3.1.5 Impact

All porous materials can crush when compressed, which suggests potential capability to absorb energy in impact. Random structured sponges have been tested, showing that plastic collapse happened at all strain rates, but with a higher fraction of cracking at high speeds, and that samples showed strain rate sensitivity, which was affected by the density, and thus the foam structure ^[123, 124] experimental studies of high speed impact of Ti lattices (**Fig. 15**) have shown that there is a protective effect, and emergent rate dependence due to the structure, indicating that design of structures could tailor the response ^[79].

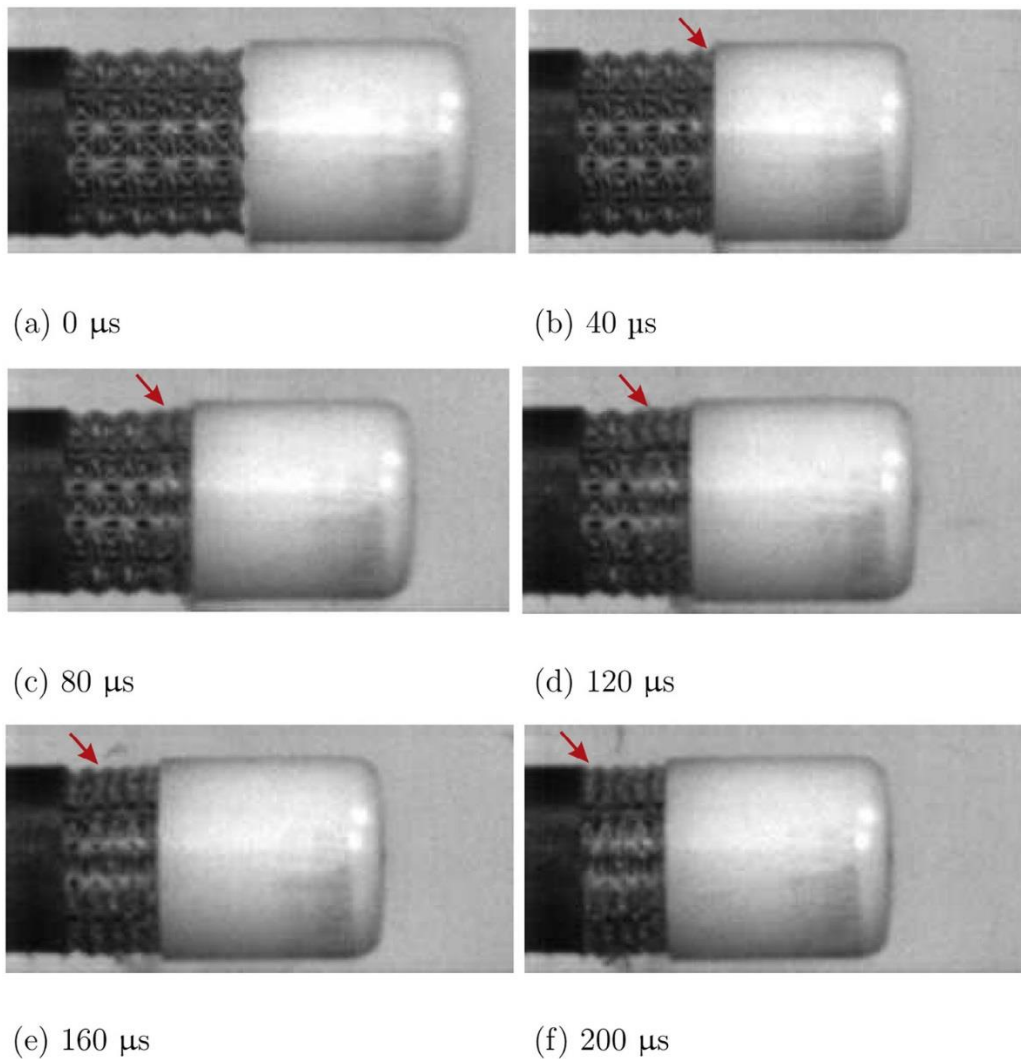


Fig. 15 – Images from high speed video of a titanium lattice under high speed impact (nylon 66 impactor fired at 104.0 m/s), showing layer by layer (inertia-dominated) failure. Reprinted from International Journal of Impact Engineering, 89, Z Ozdemir, E Hernandez-Nava, A Tyas, J A. Warren, S D. Fay, R Goodall, I Todd, H Askes, Energy absorption in lattice structures in dynamics: Experiments, 49-61, Copyright (2016), with permission from Elsevier

4. Applications

Theoretical studies exploring material selection have demonstrated that, if available in large quantities at a suitable cost, porous titanium could be attractive for lightweight structural applications ^[125].

However, examination of the dimensions produced by the methods described in section 2 shows that significant technological advances would be required for production at this scale, and many of the methods would suffer from fundamental scalability issues in reaching dimensions of the order of meters.

4.1 Biomedical Implants

Due to the small part dimensions typically required, and the relatively small volume of production, of the potential applications open celled porous titanium is often suggested for use in biomedical implants into hard tissue, for example as reviewed for dental implants in ^[126]. The advantages for such applications come from the inherent inertness in the body of titanium, combined with the availability of pores for cells to grow into and provide better fixation and the ability of a porous structure to be designed with a particular Young's modulus, below that of the dense metal (for example by adjusting the level of porosity). This latter behavior is thought to provide a potential route to avoid the stress shielding problem, where a stiff implant supports mechanical load, leading to unloaded bone around the fixation being resorbed by the body. In particular, the Additive Manufacturing techniques have been of interest, combining as they do the potential for individual tailoring of a patient-specific implant. Examples of Biomedical products making use of such technology are shown in **Fig.16.** and are commercially available including Novax DMA (www.novaxdma.com) for cranial implants, Regenerex® Porous Titanium Construct (Biomet, www.biomet.com) used in joint reconstruction or the EndoLIF® O-Cage lumbar spine fusion (Joinmax GmbH, www.joinmax.com). Porous titanium has also been investigated for drug release applications and promising results have been reported ^[127].

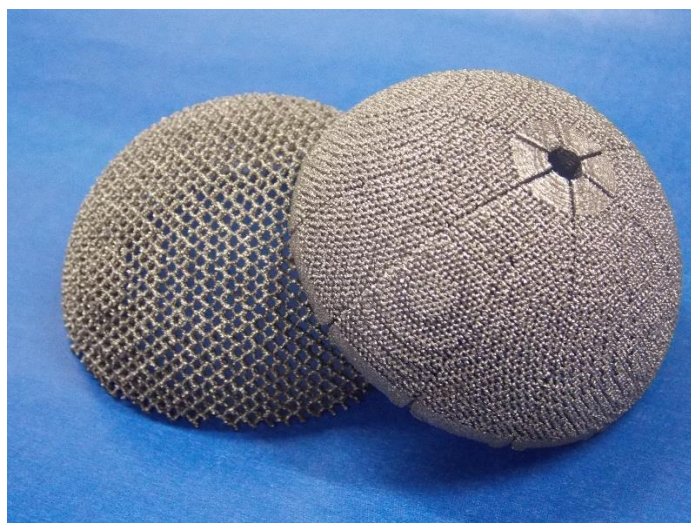


Fig. 16 – Image of Acetabular hip cup with a diameter of 57 mm. Courtesy of Sheffield University

4.2 Electrodes

Having an extremely high specific surface area makes porous Ti a potential candidate for several functional applications, including as a material for electrodes in batteries. For example, the 3C Crista Chemical Company reports the use of Ti foams in next generation lightweight car batteries, claiming that the higher active surface area of these foams leads to the energy/volume ratio being greater than lead-acid batteries, and also that batteries using titanium foams can support many more charging cycles. The manufacturing technology is likely to be electrodeposition based (US Patent 9,023,187).

4.3 Filters

The chemical and temperature resistance make porous titanium suitable for certain applications in filtration, and some Ti filters, produced through sintered powder, are already commercially available. LOFMET™ Filter Cartridges from Eaton Technologies GmbH (Nettersheim, www.eaton.de/filtration) and TPM™ Series Filter Cartridges from Graver Technologies (Glasgow, www.gravertech.com), are both sold for filtering in the size range 0.5-100 and 35µm respectively under extreme operating conditions with a maximum operating temperature of 371°C and aggressive fluids and gases.

5. Concluding remarks

The discussion in this review can be compared with that in several previous reviews of metal foam technology, such as those referred to in the introduction. One of the most striking factors is that, save the widespread introduction of Additive Manufacturing capability to permit the fabrication of porous titanium structures of a wide range of forms, there have been few if any recent radical innovations in processing. This may be reflective of the maturity of many of the technologies discussed, being rather in the phase of development for industrial application, but also shows that the AM methods are particularly well suited to this type of material (albeit that products of this form will suffer from the same limitations of dimensions and scalability that can affect AM parts of all types). It is clear that with this trajectory of development of open celled titanium towards applications will come a number of issues that are likely to be mirrored in future research directions, as the focus shifts more towards components and how to design with the materials:

Integration into parts; it is rarely possible to use a porous material on its own, and most applications will require manufacture or joining into a component of greater complexity. Depending on the processing method and the service conditions, bonding foams can be difficult, though is possible with methods like brazing ^[128], and AM techniques can create varying (or graded) structures ^[108], which could be used to not only integrate porous material with dense parts of a component, but also to vary the properties of the porous material as needed.

Microstructure control; for structural applications, as well as an increase of the size capability and cost effectiveness of manufacturing processes, there is a need for wider exploration of how the microstructure of the titanium making up the foams can be controlled by thermomechanical treatment or other methods. Typically, porous titanium is tested in whatever microstructural condition results from the processing, which is optimized for the achievement of a certain macro/mesostructure. Dense titanium alloys may undergo significant processing to achieve highly controlled microstructures and

particular optimized properties, and being able to progress towards engineered metal condition for porous titanium would help to ensure that the theoretical properties were close to being achieved.

Advances in these areas, along with further development in ensuring consistent properties, will see open celled porous titanium appearing in products across a range of areas.

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Table of Contents

Porous metals cover a wide range of materials, foams, sponges and lattices, and are of interest for some of the unusual properties and property combinations that they can possess. Here the processing,

properties and current applications of porous titanium are reviewed, highlighting the state of the art of these materials, and delineating future challenges.

