3D printing of ceramic microlattices and derived composites

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**3D PRINTING OF CERAMIC MICROLATTICES AND
DERIVED COMPOSITES**

YAP XIU YUN

SCHOOL OF MATERIALS SCIENCE AND ENGINEERING

2020

3D PRINTING OF CERMIC MICROLATTICES AND DERIVED COMPOSITES

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SCHOOL OF MATERIALS SCIENCE AND ENGINEERING

A thesis submitted to the Nanyang Technological University
in partial fulfilment of the requirement for the degree of
Masters of Engineering

2020

Statement of Originality

I hereby certify that the work embodied in this thesis is the result of original research, is free of plagiarised materials, and has not been submitted for a higher degree to any other University or Institution.

2 July 2020

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18 September 2020

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Prof Gan Chee Lip

Authorship Attribution Statement

This thesis **does not** contain any materials from papers published in peer-reviewed journals or from papers accepted at conferences in which I am listed as an author.

This thesis includes content where I received support from my supervisors and collaborators as follows:

- ❖ Professor Gan Chee Lip provided the initial project direction and vetted the thesis report.
- ❖ Advices were taken from Professor Gan Chee Lip, Dr Du Zehui, Dr Lai Chang Quan, Dr Ian Seetoh and Dr Goh Wei Liang.
- ❖ I designed the structures in Solidworks while Mr Ye Pengcheng from 3D Creatz helped with the 3D printing of ceramic structures.
- ❖ I conducted the experimental studies while Dr Ian Seetoh conducted simulation studies.

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Abstract

Mechanical properties of 3D printed ceramics with structural advantage were investigated. Three structures were studied namely, Kelvin Cell (bending dominated), Octet Truss (stretching dominated) and Simple Cubic lattice (stretching dominated). The structures were 3D printed using the stereolithography method at different relative densities of alumina.

Across the lattices, as the relative density is increased, the compressive strength, Young's modulus and energy absorption also increased. The ceramic lattices have higher compressive strength and stiffness, but lower strain and energy absorption compared to ductile lattices made of polymer or metal. Simple Cubic lattices have the best performance because of its vertically aligned struts that underwent mostly compressive forces. The inclined struts in Kelvin Cells and Octet Truss lattices induce higher tensile stresses in the joints, which does not work well for ceramic materials. Unlike ductile lattices, ceramic lattices undergo brittle fracture and have minimum densification phenomenon. The low relative density Kelvin Cells and Octet Truss lattices exhibit pseudoplastic behavior which would give users time to react before a catastrophic failure occurs.

Another study was done on the mechanical properties of Octet Truss Epoxy-Alumina Interpenetrating Phase Composite (IPC). Fabrication process for IPC was established in the study. The secondary phase provides support to the lattice and prevents catastrophic failure of Octet Truss IPC, resulting in improved specific compressive strength, strain and energy absorption. Such IPCs could potentially fill the gap for stronger structures that have higher ductility to provide better energy absorption properties per unit weight. This could be utilized in light weight shock absorption applications such as body armour and crash barriers.

Lay Summary

Additive manufacturing or 3D printing has been gaining lots of interest in recent years due to its importance to Industry 4.0. Fabricating lattices can create low weight and high strength structures. However, most of them use ductile (i.e. softer and able to deform) materials like polymers and metals which are able to withstand the stretching and bending during the loading process. Few studies have been done on ceramic lattice structures due to the inaccessibility of 3D printing technology for ceramics. The ductility of the lattice structure allows the lattice to exploit its geometrical advantage where it can bend and compress without fracturing catastrophically. Therefore, although ceramics have higher strength and stiffness compared to metals and polymers when it comes to its bulk performance, it is unclear if the same is true in lattices.

The objective of this study is to understand the performance of lattice structures made of a brittle material (e.g. alumina) using 3D printing technology, stereolithography. Additionally, a second material was added to fill the gaps within the lattice, thereby creating what is known as Interpenetrating Phase Composites (IPC). This is somewhat similar to conventional composites which mixes different materials together to obtain properties better than both of the original materials. The difference lies in that there is now full control of how the secondary material will be arranged and both components are interconnected. Producing IPC using ceramic lattice as the secondary phase has now been made possible with 3D printing of ceramics.

Mechanical tests (i.e. compression test) were done to understand the mechanical performance of the ceramic lattices and IPCs. Compared to ductile materials, ceramics did retain its higher strength and stiffness property even in the form of lattices. It was also found that due to the brittleness of the ceramic material, it is very sensitive to geometric changes. Having a geometry that underwent predominantly compressive force in the struts yielded much better performance compared to geometries where struts experienced more bending and stretching during compression. On a per unit weight, the IPCs performed better than the standalone lattices.

This study provided deeper understanding to the mechanical behaviour of brittle material lattice structures and serves as a groundwork for future ceramic lattice studies. On top of that, ceramic lattices in this study has the potential to be used as a new material for harsh environment usage i.e. high temperature and high-pressure environment.

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Chapter 1

Introduction

This chapter gives a brief overview on the development of 3D printing thus far. A brief discussion on the rationale for this study will be presented. The objectives, scope and expected outcomes for this study are also stated in this chapter.

1.1 Introduction

3D printing, a form of additive manufacturing, was developed by Charles Hull in 1986 [1]. This process prints successive layers of material on top of each other. 3D printing allows the freedom to design complex geometries and eases the manufacturing of these customized parts with high precision, which is a huge advantage as compared to traditional manufacturing methods such as slip casting and gel casting, which are unable to produce overly complex shapes such as 3D lattices. This advantage of customizing comes with a costly price tag which limits its use in many industries, especially in those which require large quantity production. It is only in recent years that new technology developments have reduced the cost of 3D printing and has been utilized in more industries, from prototyping to production [2]–[5].

With the advancement of 3D printing technology, products can be printed to suit the specific needs of the application; designs can be changed without additional cost of preparing the mold. In the biomedical field, implants can be printed to be patient-specific which helps to improve the comfort of the consumer [6], [7]. Another field of research is the 3D printing of lattice structures that can help enhance the mechanical properties of an object while reducing its density. These cellular materials are generally characterized by high specific strength, large densification strain and the possibility of a smooth plateau stress during loading. These structures are usually targeted for protective applications and have been analyzed more for their energy absorption capability [8], [9]. There have been many studies looking into various kinds of structures which range from bending-dominated to stretching-dominated. However, these studies focused on metals and polymers as the technology for printing these materials are more developed and hence, widely available [2], [10]–[15]. However, studies using ceramics are relatively less common [16].

Ceramic is known for its high strength and hardness as compared to metals and polymers. However, they also have low fracture toughness and strain. Advanced ceramics are commonly used in harsh environment applications such as automotive, aerospace and defense industry. However, fabricating advanced ceramics using traditional methods require post-hard machining using expensive diamond tools which is time consuming [17]–[21], and the geometries are usually simple ones such as

cylinders and plates [22], [23]. Now, with the development of 3D printing for ceramics, it opens up the possibility of fabricating high strength ceramics with complex structures to create high strength to weight ratio products. This pushes the boundaries of research for lightweight materials which possess high mechanical strength.

Composite materials are a well-researched topic where two materials are combined to complement each other. For example, a metal matrix composite where ceramic particles or whiskers are added as the second phase, is expected to produce materials with higher strength than the matrix metal with reasonable strain [24]–[26]. However, the second phase in most composites are not connected, that means the force transmission is through the matrix which the second phase is embedded in. That raises the question on the effect of having a composite where the second phase is interconnected with a specific structure. Simulations suggest that they may contribute towards higher specific strength of the material, with the strength and strain exceeding the component materials' properties [27]. Therefore in this study, the effects of infiltrating a second material to the lattice structure, which is known as interpenetrating phase composites (IPC), will be studied too [28], [29].

1.2 Hypothesis

We herein propose that:

1. Ceramic lattices can give higher strength to weight ratio compared to metal and polymer lattices.
2. Interpenetrating phase composites made from ceramic lattices and polymer matrix can give a higher strength and strain than each of the constituent materials.

1.3 Objective and Scope

To study the IPC of ceramic lattices, we must first understand the behavior of ceramic lattices and how they differ from other ductile materials such as polymers and metals. The objectives and scope of this study are as follows:

1. To understand the mechanical performance of 3D printed ceramic lattice structures through varying the lattice design and relative density of the lattice structure.
2. To develop a method to fabricate interpenetrating phase composite with improved mechanical properties.

1.4 Expected Outcomes

The expected outcomes of this research are as follows:

1. An analysis on the effects of ceramic lattice's topology and relative density on its mechanical performance.
2. A proof of concept that a ceramics lattice – polymer matrix IPC will have enhanced mechanical properties over its constituents.

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Chapter 2

Literature Review

In this chapter, a brief overview of 3D printing will be given first. Following that, it will present literature reviews on the types of 3D structures and the reasons behind choosing the lattice structures used in this study. Ceramics 3D printing technological and material developments will also be discussed. An introduction to interpenetrating composites (IPCs) and its current studies will be discussed. Factors influencing a structure's mechanical properties will also be analysed, and this will guide the parameters of the lattice features to be kept constant in this study.

2.1 3D Printing

This section seeks to give a brief overview on 3D printing and the common methods used before delving deeper into the main content for this study. There are three types of fundamental fabrication processes: formative, subtractive and additive; 3D printing is classified under Additive Manufacturing (AM). Figure 2-1 shows the four main components in AM: (1) input, (2) material, (3) method, and (4) application [1].

Figure 2-1: Additive manufacturing wheel depicting the four major aspects of AM [1].

The common methods of additive manufacturing are fused deposition modelling, powder bed fusion, inkjet printing, stereolithography (SLA), direct energy deposition and laminated object manufacturing. Ngo *et al.* [2] gave a summary on the materials, applications, resolution range, benefits and drawbacks of the common methods of additive manufacturing as shown in Table 2-1.

Table 2-1: Summary of materials, application, benefits and drawbacks of the common methods of additive manufacturing adapted from [2].

Methods	Materials	Applications	Benefits	Drawbacks	Resolution range (μm)
Fused deposition modelling	Continuous filament of thermoplastic polymers Continuous fibre-reinforced polymers	Rapid prototyping Toys Advanced composite parts	Low cost High speed Simplicity	Weak mechanical properties Limited materials (only thermoplastics) Layer-by-layer finish	50 – 200
Powder bed fusion (SLS, SLM, 3DP)	Compacted fine powders Metals, alloys and limited polymers (SLS or SLM) Ceramic and polymers (3DP)	Biomedical Electronics Aerospace Lightweight structures (lattices) Heat exchangers	Fine resolution High quality	Slow printing Expensive High porosity in the binder method (3DP)	80 – 250
Inkjet printing and contour crafting	A concentrated dispersion of particles in a liquid (ink or paste) Ceramic, concrete and soil	Biomedical Large structures Buildings	Ability to print large structures Quick printing	Maintaining workability Coarse resolution Lack of adhesion between layers Layer-by-layer finish	Inkjet: 5 – 200 Contour crafting: 25 – 40
Stereolithography	A resin with photo-active monomers Hybrid polymer-ceramics	Biomedical Prototyping	Fine resolution High quality	Very limited materials Slow printing Expensive	10
Direct energy deposition	Metals and alloys in the form of powder or wire Ceramics and polymers	Aerospace Retrofitting Repair Cladding Biomedical	Reduced manufacturing time and cost Excellent mechanical properties Controlled microstructure Accurate composition control Excellent for repair and retrofitting	Low accuracy Low surface quality Need for a dense support structure Limitation in printing complex shapes with fine details	250
Laminated object manufacturing	Polymer composites Ceramics Paper Metal-filled tapes Metal rolls	Paper manufacturing Foundry industries Electronics Smart structures	Reduced tooling and manufacturing time A vast range of materials Low cost Excellent for manufacturing of larger structures	Inferior surface quality and dimensional accuracy Limitations in manufacturing of complex shapes	Depends on the thickness of the laminates

To determine a suitable printing method, one must know the type of material to be printed as some methods can only print specific ones, such as fused deposition modelling can only print polymers. Application is also an important factor to consider as it determines what resolution and printing quality are required and thus, the method to be used. Across the common 3D printing methods and materials, the powder size, shape and distribution, as well as the chemistry and rheology of the medium holding the powders together, play an important role in determining the quality of the printed parts [3]–[5]. The type of laser used in the method is also important to the outcome of the part, and more information can be found in the paper written by Lee *et al.* [6] on the different types of lasers and their effects on 3D printing.

In this study, stereolithography method will be used to print the ceramic parts. Before printing, there is a standard procedure to prepare the design to be read by the 3D printing machine.

Figure 2-2 illustrates the process flow in additive manufacturing [7]. The design is first drawn using a Computer Aided Design (CAD) software (e.g. Solidworks). Subsequently, this design would be saved in .STL file format. This format encodes only the surface geometry of a 3D object through tessellation. It is used with a 3D slicer to allow a computer to communicate with a 3D printer hardware to carry out the printing [8].

Figure 2-2: Process flow in additive manufacturing [7].

With the advancement in 3D printing technology, the applications adopting this manufacturing method has grown. Recently, a research team in Nanyang Technological University (NTU) Singapore, 3D printed a bathroom, cutting the production time and cost by nearly 50% [9]. In China, cheap houses can be printed in less than a day [10]. However, the main advantage of 3D printing technologies is their ability to print

complicated structures, such as lattice structures, which cannot be fabricated using traditional manufacturing methods [11]. Designing a specific microarchitecture allows the exploitation of both the structural advantage and material strength, leading to superior cellular materials, which are known as mechanical metamaterials [12], [13]. Many lattice structures and innovative solutions, mainly for low density-high energy absorbing materials, have been developed but improvements are still necessary [2]. Such structures are required in many applications such as materials within an Earth Return Capsule [14] and protective materials [15]. Regardless the class of material, there are three dominant factors that govern their behavior: (1) material property of the foam or lattice, (2) topology and shape of the cell edges and faces, and (3) relative density (volume fraction, V_f) of the foam or lattice [16]. Figure 2-3 shows a chart on the three main factors governing the behavior of the cellular solid. The subsequent section will review the studies that have been reported on lightweight cell topologies of high specific strength and stiffness.

Figure 2-3: The 3 main factors governing the behaviour of the cellular solid [16].

2.2 3D Structures

This section will give an overview of how 3D lattices are characterized as well as discuss the studies that have been done on 3D structures.

2.2.1 Mechanical Behaviour

3D lattice structures can be classified into two main groups, bending dominated and stretching dominated of the cell walls. The Maxwell rule for a pin-jointed frame in 3D is given by Equation 2-1,

$$M = b - 3j + 6 \quad (2-1)$$

where M is the mechanism, b is the number of struts and j is the frictionless joints; this equation determines how the structure will fail. When $M < 0$ where joint connectivity is low, the failure mechanism is bending dominated and when $M > 0$ where there are more struts meeting at the joints, the failure mechanism is stretching dominated. As the name suggests, bending dominated structures fail through cell wall bending while stretching dominated structures fail through cell wall stretching. Figure 2-4 illustrates the forces acting on the struts of Kelvin Cell (bending dominated), also known as Tetrakaidecahedron, and Octet Truss (stretching dominated) unit cell under compression loading. These two structure groups have different mechanical performance. Bending dominated structures have lower structural efficiency but can take on more strain while stretching dominated structures offer greater stiffness and strength. As such, bending dominated structures are commonly used for energy absorption applications [17]–[19] while stretching dominated structures for lightweight-high strength applications [20], [21].

Figure 2-4: Schematic illustrating the force on the struts of a (a) Kelvin Cell (bending dominated) and (b) Octet Truss (stretching dominated) under compression loading [22].

According to the Ashby analysis, the relative stiffness and relative strength of both lattice groups can be approximated by a power law relationship with relative density (e.g. $E/E_s \propto (\rho/\rho_s)^P$). Figure 2-5 and Figure 2-6 show the power law for relative modulus and relative strength against relative density, respectively, for ideal bending dominated and stretching dominated structures. These values meant that stretching dominated structures are stronger and stiffer for the same relative density. However, these values are less certain for brittle materials like ceramics. This is because brittle fracture is highly dependent on defect distribution which is a stochastic process and the failure of the first strut may trigger the failure of the whole structure. Therefore, it is important to understand how brittle lattices, like ceramic, deviate from the ideal power law relationship.

Figure 2-5: Logarithmic plot of relative modulus against relative density for ductile cellular structures [16].

Figure 2-6: Logarithmic plot of relative strength against relative density for ductile cellular structures [16].

Figure 2-7 shows a logarithmic plot of compressive stiffness and compressive strength against relative density of different materials. Zheng *et al.* [22] tested solid alumina and HDDA (hexanediol diacrylate) Octet Truss (stretching dominated) and HDDA Kelvin Cell (bending dominated) with $\rho/\rho_s < 0.2$. His results for the respective mechanical properties against relative density were close to the desired ideal relationship for the respective structures. Particularly for the alumina Octet Truss lattice, the power fit law for compressive stiffness and compressive strength follow a linear relationship with relative density. However, it remains unknown if these results would extrapolate to higher relative density. Mohr *et al.* [23] did a simulation study on metallic Octet Truss for $\rho/\rho_s = 0.1 - 0.5$ and found that the fracture mechanism changes as the relative density increases. The fracture stabilizes at high relative density which led to higher strength for the lattice. Such a phenomenon renders the possibility of the power law changing with relative density since the fracture mechanism change could mean greater improvement in mechanical properties. Therefore, in this study, experiments would be conducted on a wider range of relative density ($\rho/\rho_s = 0.1 - 0.5$)

to gain a better understanding of the variation of this commonly used power fit law, in relation to relative density.

Figure 2-7: Logarithmic plot of relative compressive stiffness and relative compressive strength against relative density for stretching and bending dominated structures of different materials [22].

The stress-strain curves also differ for the stretching dominated and bending dominated structures. Figure 2-8 (a) shows the stress-strain curve of a bending dominated structure made from an elastic material [16]. The structure deforms linear elastically up to its elastic limit before yielding plastically. At the plateau region, cell wall bending occurs. On the other hand, stretching dominated lattices have a steeper Young's modulus and an initial collapse in strength (Figure 2-10 (b)). The initial drop in strength is a post-yield softening where the structure may buckle or experience a

brittle fracture which makes them less suitable for energy absorbing applications [16]. For both groups of lattices, after the plateau region, the structure densifies where the whole structure is compressed together before it eventually fails.

Figure 2-8: General stress-strain curve for (a) bending dominated structures and (b) stretching dominated structures, for ductile materials [16].

The above is true for ductile lattices like polymer and metals. However, other studies suggest that brittle materials have a different mechanical response. Mahesh *et al.* [24] did a numerical simulation on the fracture response of concrete square lattice. Figure 2-9 shows the force against displacement curve for axial forces, and axial and bending forces. It is observed that brittle lattices do not have a densification region as with the ductile lattices (Figure 2-8) and the plateau region is also at a much lower stress level than its peak stress. After the steep drop in force, the force on the lattice is seen to taper off at a much lower level, suggesting the concrete lattice had lost a good portion of its structural strength.

Figure 2-9: Numerical results for influence of failure mode on concrete square lattice [24].

The above study suggests that brittle lattices do not follow the general trend for ductile lattices. Due to the difference in ductility, ceramic lattices are likely to have a far different performance as compared to polymer and metal lattices. Unlike the ductile lattices which have been studied extensively, relatively little is known about ceramic lattices in terms of how they perform numerically, and more so experimentally. This is partly because of the limitation in 3D printing technology of ceramics in the past. Therefore, there is of much value to understand the mechanical behaviour of ceramic lattices. Hence, in this study, alumina lattices will be printed using stereolithography (SLA) process and compression test will be done on them. Simulation will also be done with a collaborator to understand the fracture mechanism of alumina lattices and how they differ from the commonly known polymer and metal lattices. The following section will review the studies that have been done on the different groups of lattices.

2.2.2 Structural Designs

Amongst a multitude of different bending dominated lattice geometries (e.g. foam cells, body centered cubic lattices, etc.), Kelvin Cells have been commonly used to represent bending dominated structures [25]. Extensive studies have been done on Kelvin Cell, both numerically and experimentally. Models have been developed to predict the structural and mechanical response under loading [26]–[31] while experiments have been done to test the mechanical performance of Kelvin Cell [19] and

compare with other geometries. Zhong *et al.* [32] compared Kelvin Cell with Diamond structure and Body-Centered Cubic (BCC) structure cell topologies using stainless steel and found that Kelvin Cell has the highest compressive and energy absorption performance. Figure 2-10 shows the simulation result of the Kelvin Cell at 50% strain. It was reported that the breaking occurred 45° to the loading direction along the red line due to the action of shearing.

Figure 2-10: Simulation result for stainless steel Kelvin Cell structure with $\rho/\rho_s = 0.4$ at 50% strain [32].

As for stretching dominated structures, Octet Truss [33] is commonly used as a representation [34]–[36]. Similarly, the work done on this structure has been extensive in both simulation and experiments. Mohr *et al.* [23] conducted a numerical study backed by experiments to understand how the fracture of stainless steel Octet Truss changes with relative density. Figure 2-11 shows the simulation results for $\rho/\rho_s = 0.05$ and $\rho/\rho_s = 0.5$. His group found that at relative density above 0.3, the structural response for compressive loading changed from an unstable twist mode to stable buckling free mode resulting in an improvement in the compressive strength. Jin *et al.* [37] obtained a similar trend where increasing the relative density of Ti-6Al-4V Octet Truss enhances its compressive strength. In terms of the fracture behaviour, Octet Truss has the highest stress intensity along the diagonal plane of the lattice as shown in Figure 2-11 (b). This pattern is identical to what was observed for Kelvin Cell [32].

Figure 2-11: Density dependent Octet Truss unit cell deformation mode: (a) twist mode at low relative densities and (b) stable mode at high relative densities [23].

Simple Cubic structure is a stretching dominated lattice but with a different fracture pattern from Octet Truss. Kadkhodapour *et al.* [38] simulation prediction for Ti-6Al-4V Simple Cubic lattice found that the struts aligned with the loading axis takes on bulk of the load and fractures layer-by-layer in the vertical struts. Analyzing the computational fracture predictions in Figure 2-12, it seems to suggest that the vertical struts in Simple Cubic structures undergo little bending and experience minimal tensile forces as compared to the Kelvin Cell and Octet Truss because of the way the struts are aligned to the loading axis.

Figure 2-12: Simulation result for Ti-6Al-4V Simple Cubic structure showing the stress intensity in the lattice during compression loading. Blue regions have the lowest stress intensity while the red regions have the highest [38].

The variety of structures that have been developed is definitely not limited by those reviewed above, there are other studies that investigated optimizing topological design to improve the mechanical response like strength and energy absorbance [39], [40]. In a review of several 3D printed structures, Oraib *et al.* found that sheet-diamond structures have the best mechanical performance compared to nine other structures as seen in Figure 2-13 [40].

Figure 2-13: Ten types of structures compared by Oraib et al. where the structures were sub-divided into three groups; (a) strut-based cellular structure, (b) skeletal-TPMS based cellular structures and (c) sheet-TPMS based cellular structures.

In essence, there are many different lattice structures that have been developed that exhibit good mechanical properties but in terms of representing bending dominated and stretching dominated mechanism, among those, Kelvin Cell and Octet Truss are most widely used, respectively.

2.3 Geometric Factors

This section will review the geometric factors that influence the performance of the lattice; this includes the strut shape and size, and the structure size.

2.3.1 Strut Shape and Size

Previous studies have shown that for brittle materials like ceramics, compressive fracture strength typically increases with decreasing dimensions [41], [42]. Based on the relationship between fracture strength and critical flaw size proposed by Griffith [43] and assuming that critical flaw size correlates with the strut thickness, t [44], we have the relationship as represented in Equation 2-2.

$$\sigma_f \propto t^{-0.5} \quad (2-2)$$

This suggests that as strut thickness decreases, the fracture strength will increase. Cui *et al.* [45] did a study on the effect of strut size on the strength of lattices using struts with squared cross section. The study found that at a fixed relative density and strut length, as strut thickness increases, the normalized strength would decrease. In addition, as the volume of strut increases, the normalized strength would also decrease.

With this, the diameter designs in this study will be kept to the smallest possible size attainable for the paste and 3D printing machine. To reduce the possible stress concentration due to sharp edges, cylindrical struts of diameter 0.5 mm will be used. To decrease the relative density, the strut length will be increased while keeping strut diameter constant.

2.3.2 Structure Size

Structure size refers to the number of unit cells in a lattice. A study by Lai *et al.* [46] correlated the number of unit cells in a structure with structural strength, stiffness and energy absorption. There are two types of size effects as shown in Figure 2-14: (a) lateral size effect, and (b) along-the-stress size effect [47].

Figure 2-14: Two types of size effects: (a) Lateral size effect and (b) Along-the-stress size effect [47].

L. Yang [47] did a simulation study on the structure size and aspect ratio effect on various structures (i.e. re-entrant auxetic, Octet Truss, rhombic and BCC lattice) using Ti6Al4V. The study found that increasing lateral size has a positive impact on the mechanical properties while along-the-stress size has a negative effect. For an aspect ratio of 1:1, the normalized strength would increase as the structure size increased. An interesting thing to note is that for larger Octet Truss structures where there are more than eight unit cells in each direction, the initial failure strength was observed to reduce by 60%. Comparing different studies on this topic [46]–[50], it is observed that different topologies and materials can result in varying sensitivity of the mechanical properties to structure size. One common trend is that for a structure of 1:1 aspect ratio, the normalized strength would increase with the number of unit cells. However, exceeding a certain number of unit cells, the maximum strength will not increase with increasing unit cells. This number is structure dependent.

In view of the above finding on effect of structure size, the structure size would be kept constant for all test samples in this study to minimize the influence by structural size. Dimension limitations from the 3D printing machine used in this study would also have to be considered. Hence, the structure size will be kept at 4x4x4 unit cells.

2.4 Ceramics 3D Printing

Ceramics 3D printing technology has been investigated in recent years, allowing fabrication of ceramics with complex geometries. With their high mechanical strength and hardness as well as excellent chemical and thermal stability, the

availability of ceramics 3D printing technology would offer new possibilities for ceramics applications, such as the biomedical [51], [52], aerospace and electrical fields [53]. This section will discuss ceramic 3D printing technologies and developments in printable ceramic materials.

3D printing technology can be classified according to their feedstock form, i.e. slurry, powder and bulk-solid based. A slurry-based process starts off with a polymer paste containing fine ceramic particles that have a range of viscosity depending on the system. These are polymerization-based techniques which involve curing the polymer using different light source (depending on the technique) according to the desired pattern before post-processing (i.e. debinding and sintering) to obtain the ceramic product. Examples of these slurry-based techniques are stereolithography (SLA) [54], digital light process (DLP) [55] and two-photon polymerization (TPP) [56]. Inkjet-based printing (IJP) and extrusion-based direct ink writing (DIW) are slightly different slurry-based techniques in that they build the part droplet by droplet instead of polymerizing the pattern on a bed of ceramic paste. Powder-based techniques include three-dimensional printing (3DP) [57], selective laser sintering (SLS) [58] and selective laser melting (SLM). 3DP selectively spreads liquid binders on a bed of loose ceramic particles while SLS and SLM use thermal energy from the laser beam to sinter or melt the ceramic particles, respectively. Bulk-solid based techniques include laminated object manufacturing (LOM) [59] and fused deposition modelling (FDM) [60], [61]. LOM process involves pre-cut thin sheets of ceramic pre-coated with adhesive and stack over one another. Heating is done to bond the layers together to form the 3D product. On the other hand, FDM uses pre-made ceramic-polymer composite filament to 3D print the part which is subsequently debinded and sintered to achieve densification. Of these techniques, slurry-based photopolymerization methods (i.e. SLA, DLP and TPP) have greater potential due to its high controllability in the printing specifications and mechanical performance.

The above ceramic 3D printing methods are able to print many types of ceramics, some of which include Al_2O_3 , SiO_2 , ZrO_2 and SiC . Other than printing singular solid materials, technologies have also improved to 3D print hollow ceramic lattice ceramic [62] and ceramic composite materials (e.g. ceramic-metal composite). Some studies have explored hollow ceramic lattices which have shown some promising

results in terms of lowering the density while maintaining reasonable mechanical strength and stiffness [21], [22]. Figure 2-15 shows the fabrication process of titanium nitride hollow ceramic lattice. The process uses a polymer material as a scaffold to coat the ceramic material using atomic layer deposition before creating a small gap to facilitate removal of the polymeric material within through melting procedures such as plasma etching.

Figure 2-15: Schematic of fabrication process for hollow ceramic lattice taken from [62].

In a more recent development, thermoplastic 3D printing has also made it possible to print ceramic-metal composites. Scheithauer *et al.* [63] combined fused deposition method (FDM) and robocasting to 3D print steel-zirconia composites. They used a thermoplastic suspension of relatively low viscosity (suitable for high pressure injection molding of ceramics) to allow the feedstock to pass through a thin nozzle. The stainless steel and zirconia feedstock were stored in two separate dosing unit as shown in Figure 2-16. To print the composite, stainless steel and zirconia were printed separately on the same layer, resulting in stainless steel being embedded within the zirconia structure. With this additive manufacturing method, other material combination may also be printed provided both components can be co-sintered.

Figure 2-16: Schematic illustrating thermoplastic 3D printing for two materials [63].

2.5 Interpenetrating Phase Composites (IPC)

Figure 2-17 shows a Young's modulus - density material property chart. The coloured bubbles represent property range for family of materials and the small white bubbles within the larger bubbles represent individual materials. The orange arrow pointing to the top left defines a vector for material development; if the hole at the top left of the chart can be filled, it will enable lighter and stiffer materials [64]. Mechanical metamaterials have potential to fill these areas in the sense that they can tap on the structural and material strength to reduce its density, thereby increasing the specific mechanical property.

Figure 2-17: Modulus-density material property chart showing areas occupied by present materials and potential spaces that could be occupied by hybrid materials [64].

To improve the property of a material, a second component is usually added. This new hybrid material is commonly known as a composite. Many composites are fabricated through the addition of a second phase in powder or whisker form that would help to improve the composite properties. However, one major drawback of powder / whisker enhanced composite is its random and isolated arrangement, which limits its effectiveness in improving the matrix properties. Sun *et al.* [65] found that random particle arrangement causes stress concentrations as compared to periodic powder arrangements. Interconnectivity is also an important multifunctional characteristic in applications such as electrical and structural [66], [67].

Hence, this is where interpenetrating phase composites (IPCs) can come into play. Instead of single structures, the structures may be filled with a second phase to obtain a solid material with a continuous, periodic network throughout the matrix

material. The matrix material could act as an additional support to the bending or stretching dominated structures, making them resistant to bending and buckling loads, thus, enhancing the strength of IPCs. There are several studies on infiltrating foams with a molten matrix [68], [69]. However, this concept of infiltrating specific periodic structure is still relatively new. Figure 2-18 illustrates the concept of IPCs where the periodic structure is infiltrated with a matrix material [70]. Oraib *et al.* [70] studied the compressive behaviour of polymer-based IPCs and found that the matrix (softer phase) confines cracks and prevents catastrophic failure while the lattice (harder phase) endures a larger fraction of the load. Seetoh *et al.* [71] also experimented on polymer-based IPCs. In addition to the findings Oraib *et al.* obtained, he identified that the presence of the matrix also helps to distribute the load in the structure.

Figure 2-18: Schematic illustrating IPCs - (a) lattice structure, (b) lattice structure infiltrated with a matrix (IPC), (c) cross-section of IPC [70].

Applying the knowledge from Oraib's experiments, ideally, if the hard phase bears most of the load, then ceramics having higher hardness and stiffness, will be able to create IPC with good loading capability while having a ductile matrix to support and prevent catastrophic failure. Metal-ceramics composite is one of the typical adopted combinations for high strength applications [72]–[76]. Of these, alumina-aluminium composite is the most common. In one of the alumina-aluminium IPC studies, Marchi *et al.* [77], [78] printed an alumina tower structure as shown in Figure 2-19 and infiltrated it with aluminium using gas pressure infiltration. The rod diameter was 250 μm and had nominal dimensions of 6 x 4 x 4 mm^3 . The compressive strength of the IPC improved by almost a factor of 4 over the un-infiltrated tower. Their explanation is that the aluminium prevented the buckling of the load-bearing alumina columns.

Figure 2-19: Side-view of 3D printed architecture and (b) schematic of 3D tower architecture [77].

Therefore, this study seeks to examine ceramic lattice IPC in terms of its strength, stiffness, strain and energy absorbance. For a start, the lattice material will be alumina and the matrix will be epoxy. With the study of these new lattice structures and IPC materials, it could potentially produce a lightweight, high strength and tough material to fill a part of the hole in the material property chart as shown in Figure 2-17.

2.6 Conclusion

In majority of the studies done, including those that have not been reviewed here, softer and tougher materials like metals or polymers were used. These materials are ductile enough to undergo bending which allows the structure to absorb more energy, resulting in a longer plateau region in their stress-strain curve. However, few studies used solid ceramic materials for these structures. Studies that touch on 3D printed ceramics discuss more on ultralight hollow ceramic struts [21], [22] or coating of ceramics on a tougher material [79]. Unlike metals and polymers, ceramics are known for its high strength and hardness but low toughness. Therefore, it may not perform as well in structures that have high bending and tensile forces despite its superior intrinsic property. One of the aims of this study is to investigate how ceramics will perform as a printed structure as compared to ductile materials. This will be done by comparing the performance of 3D printed alumina structures with other studies that used ductile lattices. Another area of study is the influence of relative density of 3D printed ceramics on its mechanical strength. A systematic study will be done to

understand the relationship between relative density and specific strength, stiffness, failure strain and failure energy absorbance for the alumina material used in this work.

Current studies have shown that the matrix material in IPC helps in providing support to the lattice by confining the cracks in the lattice and aiding load distribution throughout the IPC. However, many of the IPC studies deal with ductile materials like polymers or metals which are not as strong and stiff as ceramics. With ceramic being such a brittle material, introducing a secondary material could help to prevent the brittle failure of ceramics, allowing a stronger and stiffer scaffold in the IPC, thereby improving its mechanical properties compared to its more ductile counterparts. Therefore, ceramic-epoxy IPC would also be experimented in this study.

In terms of studying the behaviour of structures, extensive studies have been done on Kelvin Cell (bending dominated) and Octet Truss (stretching dominated) as reviewed in the previous sub-section. Kelvin Cell and Octet Truss have shown to be an ideal model to represent these two groups of structures and they are isotropic. To compare bending and stretching dominated lattice, Zheng *et al.* [22] used Kelvin Cell and Octet Truss to represent these two groups of lattices. Szczurek *et al.* [80] also used Kelvin Cell, Octet Truss, Body-Centred Cubic (BCC) structure and Hexagonal Honeycomb to study the effects of printing paste variation. Hence, for a start, these two structures will be used to analyse the performance of ceramic in bending and stretching dominated structures. This aids future comparison with other materials due to the extensive studies done on these two structures. As reviewed above, Simple Cubic structure undergoes a different fracture mechanism (predominately compression) than many other lattices. Based on the above information, it is hypothesised that alumina Simple Cubic lattice would perform well compared to Kelvin Cell and Octet Truss since ceramic is stronger in compression than tension. Therefore, Simple Cubic lattice will also be studied in this work. Lastly, simulation studies will be done in collaboration with Dr. Lai Chang Quan and Dr. Ian Seetoh to understand the fracture mechanism of ceramic lattices.

In conclusion, the objectives and scope of this study are as follows:

- ❖ To understand the influence of topology and relative density on the mechanical behavior of ceramic lattices. Kelvin Cell, Octet Truss and Simple Cubic

structures will be printed and tested to analyze their compressive strength, stiffness, failure strain and energy absorption performance. In collaboration with another team, simulation will be done to understand the fracture mechanism of ceramic lattices.

- ❖ To develop an IPC using alumina infiltrated with epoxy; with the aim to fill a gap in the stiff and lightweight area of the material property chart shown in Figure 2-17.

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Chapter 3

Experimental Methodology

This chapter will discuss the sample preparation process, various material characterizations of 3D printed ceramics, as well as simulation of different structures. The first section will discuss the design of 3D structures, method used for 3D printing of ceramics, as well as the fabrication process for Interpenetrating Phase Composites (IPC). The second section will discuss the experimental methods used to characterize various properties of 3D printed ceramics. The third section will discuss the simulation works on the strength of various lattice structures.

3.1 Fabrication of 3D Ceramic Structures

3.1.1 Design of 3D Structures

SolidWorks software was used to design the 3D structures to be printed. To understand the sintering temperature effect on the 3D printed ceramic's mechanical strength, cylindrical rods with aspect ratio 1:2 ($\varnothing = 4$ mm and height = 8 mm) according to ASTM standard C773-88 [1] were fabricated. Figure 3-1 shows the Computer Aided Design (CAD) drawing of the 3D printed cylindrical rod. The reason for using a rod structure instead of a lattice structure was to omit the influence of structure on the intrinsic mechanical strength of the printed ceramics. The first phase of the study will investigate the sintering temperature influence on the mechanical strength and density of 3D printed alumina to choose an optimal sintering profile before proceeding with lattice design study.

Figure 3-1: CAD drawing of the 3D printed cylindrical rod.

Based on the reviewed literatures as discussed in Chapter 2, Kelvin Cell lattices (bending dominated) and Octet Truss lattices (stretching dominated) were chosen in this study. In addition, Simple Cubic lattice (stretching dominated) was also studied due to its different strut orientation from the other two structures. Kelvin Cell and Octet Truss lattices have struts angled to the loading axis while the struts in Simple Cubic lattices are either perpendicular or parallel to the loading axis. Figure 3-2 shows the unit cell of the Kelvin Cell, Octet Truss and Simple Cubic structure drawn using SolidWorks.

Figure 3-2: CAD drawing of (a) Kelvin Cell, (b) Octet Truss and (c) Simple Cubic unit cell.

According to a simulation study by Yang L. [2], changing the number of unit cells in the lateral and along-the-stress direction will cause a change in the lattice property. Hence, to eliminate the influence by the number of unit cells, the number of unit cells for compressive test was fixed at $4 \times 4 \times 4$, that is 64 unit cells per sample. In terms of lattice size, the lattices were designed according to the smallest printable dimensions, reason being with a larger lattice, there is a higher chance of the sample containing more defects. Therefore, the strut diameter was fixed at 0.5 mm, which is the smallest dimension printable for alumina while the strut length was reduced to increase the relative density (ρ/ρ_s). With the number of unit cells kept constant, the lattice dimensions varied with different relative densities.

Table 3-1 shows the geometric parameters used in this study for each lattice. Relative density of Kelvin Cell could not be increased beyond 0.3 as it would become a closed cell geometry. These structures were loaded under compression to study the effect of relative density and topology on the mechanical properties of ceramic lattices.

Table 3-1: Geometric parameters used in this study.

	Strut Diameter (mm)	Strut Length (mm)	Overall Length x Breadth x Height (mm x mm x mm)	Relative Density
Octet Truss	0.5	3	17.5 x 17.5 x 17.5	0.15
		2.5	14.7 x 14.7 x 14.7	0.20
		2	11.8 x 11.8 x 11.8	0.30
		1.75	10.4 x 10.4 x 10.4	0.40
		1.5	8.98 x 8.98 x 8.98	0.50
Kelvin Cell		1	11.7 x 11.7 x 11.7	0.15
		0.8	9.54 x 9.54 x 9.54	0.20
		0.7	8.40 x 8.40 x 8.40	0.30
Simple Cubic		2.5	10.5 x 10.5 x 10.5	0.10
		1.8	7.70 x 7.70 x 7.70	0.20
		1.35	5.90 x 5.90 x 5.90	0.30
		1.15	5.10 x 5.10 x 5.10	0.40
		0.95	4.10 x 4.10 x 4.10	0.50

The completed CAD drawings were converted to .STL files. This format encodes only the surface geometry of a 3D object through tessellation. It is used with a 3D slicer which slices up the structure into many horizontal layers before uploading it to the 3D printer, which would then print the structure in accordance to these layers [3].

3.1.2 Stereolithography (SLA)

Stereolithography was used to 3D print ceramics in this study. Figure 3-3 shows the set-up of the Ceramaker 900 3D printing machine [4] used in this study and a schematic illustrating the stereolithography process. UV laser of wavelength 355 nm was used to cure the photopolymer resin layer by layer. The mirror directs the laser in the X and Y directions according to the pattern of each layer. Each polymerized layer was about 125 μm . After each layer has been cured according to the pattern, the printing platform moved downward by 30 μm while the feeding piston moved upward to push more paste out of the paste tank. The blade then swept across the platform to spread the paste across the printing area evenly. The distance between the blade and platform was 200 μm . This allowed an over-polymerization of about 85 μm to create bonding between each layer, thus strengthening the structure. The printing speed for one part is 0.03 mm per minute. 3DCeram alumina [5] paste was used for printing; the shrinkage factors applied for this paste in X/Y/Z direction were 1.151/1.142/1.182 respectively.

The bottom layer for each structure was printed to be thicker to make the structure stronger. This is necessary to reduce the possibility of the blade sweeping the bottom part away during the spreading step, which would otherwise destroy the whole printing structure.

Figure 3-3: (a) Ceramaker 900 3D printer set-up and (b) schematic of stereolithography process.

After printing, the specimens were cleaned to remove the unpolymerized paste to reveal the 3D structure. Pressurized argon or nitrogen gas mixed with Ceraclean solution was used to clean the specimens. As the green body structure was very weak at this step, care had to be taken to prevent fracture during the cleaning process. Figure 3-4 shows a Kelvin Cell lattice before and after the first round of cleaning. Lattices had to be cleaned thoroughly to ensure no unpolymerized paste was left on the sample, or else they would be sintered with the structure and would result in additional bumps on the final lattice.

Figure 3-4: Kelvin Cell lattice: (a) Before and (b) after first round of cleaning the unpolymerized paste.

3.1.3 Post Processing of 3D Ceramics Structures

Specific details of the debinding and sintering procedures are proprietary. The following paragraph will discuss general information that can be disclose. After cleaning, the specimens were debinded using Thermoconcept ROC 150/610/16 High Temperature Tube Furnace to remove the polymer within the structure before sintering. Sintering was done in Thermoconcept HTL 20/17 Furnace. The suggested sintering temperature for alumina by 3DCeram is 1700°C. To study the effect of sintering temperature on mechanical strength of 3D printed alumina, the temperature was reduced by 50°C and 100°C from the suggested sintering temperature. The cylindrical rods were sintered at three different temperatures: 1600°C, 1650°C and 1700°C. The specimens sintered at 1700°C gave the best properties and was used as the sintering temperature for the rest of the alumina lattices.

3.2 Fabrication of Interpenetrating Phase Composites (IPCs)

Epoxy was used as the matrix in this study to investigate the effects of a second phase filling the gaps in the lattice structure; this is known as Interpenetrating Phase Composites (IPC). To prepare the epoxy, Araldite LY5052 and Aradur 5052 [6] were mixed in a ratio of 1:38 by weight. This mixture ratio was optimized in an earlier study to give an average compressive strength of 223 MPa and average Young's modulus of

1.53 GPa. Both components were mixed at 1160 rpm under 0.3 kPa vacuum for 3 minutes followed by placing under vacuum for two cycles of 4 minutes, bringing the total degassing duration to 8 minutes to remove air bubbles within the mixture. The epoxy was then poured into a polydimethylsiloxane (PDMS) mold to fill $\frac{3}{4}$ of the cavity before very slowly inserting the lattice structure into the mold to reduce the potential of inducing air bubbles into the epoxy matrix. Figure 3-5: Schematic illustrating fabrication process of epoxy IPC. illustrates this fabrication process.

Figure 3-5: Schematic illustrating fabrication process of epoxy IPC.

The IPC was cured at room temperature for 12 hours followed by 100°C for 4 hours. The sides of each IPC were ground to ensure even and flat surfaces ready for Instron compression test. Figure 3-6 shows an epoxy-alumina Octet Truss IPC (E-AOT) and an alumina Octet Truss lattice for comparison.

Figure 3-6: (a) Epoxy – alumina Octet Truss lattice IPC and (b) alumina Octet Truss lattice with $\rho/\rho_s = 0.3$.

The following IPC samples in Table 3-2 were fabricated to study the effects of adding a second phase in the 3D lattices. Only lattices with $\rho/\rho_s = 0.3, 0.4$ and 0.5 were fabricated, as lower relative densities would cause cracks in the matrix material after post heat treatment due to difference in the coefficient of thermal expansion (CTE).

Table 3-2: Lattice structures used for fabricating IPC samples.

Matrix/lattice material	Structure	ρ/ρ_s
Epoxy/alumina	Octet Truss lattice	0.3, 0.4 and 0.5

3.3 Materials Characterizations

3.3.1 Scanning Electron Microscopy (SEM)

To analyze the grain size, the specimens were coated with gold using JEOL JFC-1600 Auto Fine Coater at 20 mA for 40 seconds to obtain a coating thickness of 10 nm. Nova 600 NanoLab DualBeam SEM/FIB system was used for SEM analysis. Grain boundary intersection count method according to ASTM standard E112-10 [7] was used to measure the average surface grain size for each specimen. SEM was also used to analyze the surface morphology and original fracture surfaces.

3.3.2 Density Measurement

Density measurement for the solid cylindrical rods and IPC samples was done following the Archimedes principle. This principle states that when an object is submerged in a fluid, it experiences a buoyant force with a magnitude equivalent to the weight of the fluid displaced by the object [8], [9]. Measuring the weight of the object in air and having the values for fluid density and weight of displaced fluid by the fully submerged object, applying it to Equation 3-1, the density of the object can be calculated.

$$\frac{\text{Density of object}}{\text{Density of fluid}} = \frac{\text{Weight of object}}{\text{Weight of displaced fluid}} \quad (3-1)$$

Figure 3-7 shows the electronic densimeter used to measure the density of the cylindrical rod specimens. The Perspex box covering the machine minimized the influence of external environment on weight measurements. To calculate the relative density of the 3D lattices, the mass was divided by the edge-to-edge volume of the lattice.

Figure 3-7: Electronic densimeter used to measure density.

3.3.3 Quasi-static Compression Test

Compression test determines the material's behavior under compressive loads. Compression tests for the cylindrical rods and lattices were conducted using Instron 5567. At least 5 specimens were tested for each condition to obtain an average result. Figure 3-8 shows the bearing plates pasted on the compression plates. Tungsten carbide bearing plates ($\varnothing = 20$ mm, thickness = 3 mm) were attached to the top and bottom platen of the Instron press for the test of the cylindrical rods only. This is to prevent the yielding of the compression plates through the distribution of load. Bearing plates were not used for the 3D lattices and IPC samples because they would not cause yielding of the compression platens.

Figure 3-8: Set-up of bearing plates on platens for compression test of alumina cylindrical rods.

To analyse the fracture behaviour of lattice structures and IPC samples, high-speed videos of the compression tests were taken. The camera model was FASTCAM Mini AX200 from Photron, Japan. Figure 3-9 illustrates the test set-up where the light source and high-speed camera were placed approximately 60 cm away from the Instron tester. The video was taken at 500 fps with 1/1000 exposure time and 214 frames. The video resolution was 256x256 pixel.

Figure 3-9: Schematic of set-up for Quasi-static compression test.

For the cylindrical rods, 30 kN load cell was used. Preload of 25 N was applied to the specimen to ensure good contact between the samples and the loading plates

before starting the test. Strain rate for actual test was set at 0.001 s^{-1} and loaded until fracture. For lattice structures, 10 kN load cell was used. Preload of 1 N was applied for both the pre-test and actual test. Strain measurement was taken directly from the crosshead displacement and compliance correction was done for the machine. During the loading, the sample and machine parts experience the same force, but the displacement measurement is a combination of the displacement from the sample and the machine parts. Therefore, to determine the compliance correction, the machine was also loaded without any sample between the plates to obtain the displacement of the machine. Figure 3-10 shows the machine compliance graph. There is a transition point at 300 N, hence, compliance correction for these two regions follow a different equation. For force $> 300 \text{ N}$,

$$\text{Machine extension} = 1.6868 \times 10^{-5} \times \text{force applied} + 2.6241 \times 10^{-2} \quad (3-2)$$

Subtracting the machine extension from the measured compressive displacement will give the corrected compressive displacement.

Figure 3-10: Machine compliance graph.

Figure 3-11 shows a typical compressive stress-strain curve for bulk ceramics where the material undergoes a brittle fracture. From the plot, the Young's modulus, compressive strength, compressive strain and energy absorption can be obtained.

Figure 3-11: A typical compression test result plot for bulk ceramics.

3.4 Simulation

Simulation studies were done in collaboration with Dr Lai Chang Quan and Dr Ian Seetoh to understand the behavior of alumina lattices under compression.

Finite element analysis was performed on the lattice models using the Abaqus CAE software package together with the dynamic explicit solver. The “non-linear geometry” option was selected for these numerical simulations. Each model geometry was a 2 x 2 x 2 lattice of length L , positioned where one of the corners coincided with the origin ($x = y = w = 0$) and (u, v, w) being the corresponding nodal displacements. Symmetry boundary conditions were prescribed at the X, Y and Z faces (i.e. $u|_{x=0} = v|_{y=0} = w|_{z=0} = 0$) to replicate the behavior of a 4 x 4 x 4 lattice. In this way, the solution was achieved in a more computationally efficient manner.

The lattice models were meshed using quadratic tetrahedral 3D stress elements with characteristic lengths smaller than 1/3 of the strut diameter. Quasi-static uniaxial compression with strain ε_c was performed by prescribing displacement of $[u = 0, v = \varepsilon_c L, w = 0]$ at $y = L$, along with a ‘smooth-step’ amplitude function that gradually increased the strain rate from 10^{-7} s^{-1} to 10^{-1} s^{-1} . Compared to linear ramps commonly used elsewhere, the smooth ramp used here was able to reproduce quasi-static

conditions using a much shorter simulation time. Stress-curves were obtained by measuring the reaction forces during the compression step.

The model was assigned an isotropic elastic material with Young's modulus = 318 GPa and Poisson's ratio = 0.242. The former was based on uniaxial compressive tests on a bulk sample of alumina, while the latter was based on the technical specifications obtained from the manufacturer. The "Brittle Cracking" model in Abaqus was used to simulate the failure behaviour, based on the Rankine criterion, where cracks in brittle materials like concrete and ceramics start to form when the maximum principle tensile stress exceeds the material tensile strength, which was assigned a value of 300 MPa for alumina based on flexural tests (three-point bending). These tests also showed that bulk alumina failed very abruptly with very little post-failure characteristics. Accordingly, the post-failure direct stress and the shear retention factor were configured to fall rapidly to zero (i.e. by setting their corresponding post-failure strains to ~ 0). Element deletion was activated to facilitate visualization of failed regions and to prevent excessive local deformation speeds from interrupting the explicit solver prematurely.

3.5 References

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Chapter 4

Results and Discussion

This chapter will present and discuss the experimental results obtained in this project. This includes the effects of sintering temperature on the mechanical properties of 3D printed solid alumina and the effects of topology, relative density on the mechanical properties of 3D printed lattice structures. The study on mechanical performance of Interpenetrating Phase Composites (IPC) using epoxy matrix with alumina Octet Truss will also be presented.

4.1 Effects of Sintering Temperature on 3D Printed Alumina

Experiments in this section were conducted using 3D printed alumina rods. Figure 4-1 shows the compressive stress-strain curve of a 3D printed alumina rod sintered at 1700°C. The curve is comparable to other solid ceramic samples fabricated by conventional methods. The curve has a smooth initial increase until the peak stress is reached before a sudden drop where the ceramic experienced a brittle, catastrophic failure.

Figure 4-1: Compressive stress-strain curve of 3D printed alumina cylindrical sample sintered at 1700°C.

Table 4-1 shows the properties of 3D printed alumina at different sintering temperatures. Porosity is defined as $1 - \text{relative density}$. Figure 4-2 plots the relationship of the alumina density and average surface grain size with sintering temperature. There is a direct relationship between the density and the sintered temperature when it increases from 1600°C to 1700°C. On the other hand, for the average surface grain size of alumina, there is not much difference between 1600°C and 1650°C and it only increases significantly after 1650°C.

Table 4-1: Density, porosity, average surface grain size and compressive strength of 3D printed alumina sintered at different temperatures.

Sintering Temperature (°C)	Density, ρ (g/cm ³)	Porosity (%)	Average Surface Grain Size (μm)	Compressive Strength (GPa)
1600	3.37 ± 0.08	15.0	1.51 ± 0.24	1.48 ± 0.18
1650	3.60 ± 0.08	10.0	1.42 ± 0.19	2.02 ± 0.07
1700	3.75 ± 0.07	6.00	2.39 ± 0.45	2.29 ± 0.32

Figure 4-2: Plot of alumina density and average surface grain size against sintering temperature.

Figure 4-3 shows the SEM images of 3D printed alumina surfaces at 5000x magnification sintered at different temperatures. From the images, alumina sintered at 1700°C is observed to have the least pores between grains and the largest average grain size.

Figure 4-3: SEM images of 3D printed alumina surfaces sintered at different temperatures: (a) 1600°C, (b) 1650°C, (c) 1700°C.

Figure 4-4 shows the compressive strength of alumina under different sintering conditions. The average compressive strength increased from 1.48 ± 0.18 GPa when sintered at 1600°C, to 2.02 ± 0.07 GPa and 2.29 ± 0.32 GPa when sintered at 1650°C and 1700°C, respectively. With reduced porosity as the sintering temperature is increased, this in turn leads to an increase in compressive strength.

Figure 4-4: Compressive strength of alumina rods sintered at different temperatures.

Under these sintering conditions, it is likely that alumina underwent solid phase sintering where mass transport occurs mainly by volume and grain boundary diffusion.

This leads to a reduction in porosity and hence, densification of alumina [1]. The diffusion rate is exponentially dependent on temperature via the Arrhenius equation:

$$D = D_o \exp\left(\frac{-Q}{RT}\right) \quad (4-1)$$

where D is the diffusion coefficient, D_o is the constant, Q is the activation energy, R is the Boltzmann constant and T is the temperature. According to **Error! Reference source not found.**, as the temperature increases, the diffusion coefficient increases. This leads to an increased diffusion rate and reduced porosity, hence higher density of alumina [1]–[4].

There are two competing mechanisms which control the mechanical strength of ceramic, namely densification and grain growth. Figure 4-5 illustrates the dominant strength control mechanism of sintering process at different temperature range. At lower sintering temperature, densification process is the main mechanism which contributes to improved compressive strength. In our printed alumina, grain growth is not observed between 1600°C and 1650°C as seen in Figure 4-2. As sintering temperature or time increases, densification continues while grains also start to grow via neck formation as the pores shrink or are removed. At higher temperature range, there will be significant grain growth which will result in decrease compressive strength. As grain size increases, there will be lesser grain boundaries to inhibit dislocation movements, hence reducing the compressive strength [5]–[7]. At an optimal temperature where highest compressive strength is attained, the ceramic is fully dense with minimal grain size [4].

Figure 4-5: Relationship between sintering temperature/time and compressive strength of ceramics.

Figure 4-6 shows a schematic of the three stages in the sintering process [8]. At the initial stage of sintering, densification is the main mechanism. As sintering temperature or time increases, densification continues while grains also start to grow via neck formation as the pores shrink or are removed; this is the intermediate stage. This continues until an optimal point where highest compressive strength is attained. Past this point, the sintering process enters the final stage where densification rate slows down and grain growth accelerates, this final stage is grain growth controlled [4]. This is in agreement with the work by Chen and Wang, where they sintered Y_2O_3 and they measured the porosity and its grain size during sintering [9]. The trend is seen in Figure 4-7, where rapid grain growth was observed at the later stage of densification where the grain size increased from 150 nm to 400 nm when the relative density increased from 90% to 100%, which is a phenomenon that has been observed in many other studies [10]–[13].

Figure 4-6: Illustration of sintering process [8].

Figure 4-7. Plot of grain size of Y_2O_3 against relative density [9].

In general, porosity in ceramics has a greater influence on its mechanical properties [1]. Therefore, the higher density of alumina sintered at 1700°C has a stronger influence over the increase in grain size on the compressive strength. Moreover, at 1700°C, the sintering temperature might not have reached the

grain-growth controlled region as shown in Figure 4-5. A study on the relationship between sintering temperature and porosity of alpha alumina revealed that the density started to stabilise at temperatures above 1710°C, below which, it was still in the densification-controlled phase [14]. Thus, subsequent 3D printed alumina lattices were sintered at 1700°C which achieved the highest compressive strength among the three sintering conditions.

An alumina grade of 99.5% relative density has a reported density and compressive strength of about 3.98 g/cm³ and 2.5 GPa [15]. This is fairly close to our 3D printed alumina sintered at 1700°C where the average compressive strength is 2.29 ± 0.32 GPa with relative density of 96.4%. This shows the potential of using 3D printing, a relatively new method, for fabricating ceramics of complex shapes.

4.2 Lattice Structures

Figure 4-8 (a) – (c) shows the CAD drawing of the 4x4x4 Octet Truss lattice, Kelvin lattice and Simple Cubic lattice with $\rho/\rho_s = 0.3$. Figure 4-8 (d) – (f) shows the actual corresponding 3D printed alumina lattices. Figure 4-8 (c) also illustrates where the top and side faces are relative to the printing direction. Table 4-2 shows the designed and actual dimensions of the lattice structures. The lattice dimensions are close to the designed parameters.

Table 4-2: Designed and actual dimensions of lattice structures.

Lattice	Relative Density	Designed Dimensions (mm)	Actual Dimensions (mm)
Octet Truss	0.15	17.5 x 17.5 x 17.5	17.5 x 17.5 x 17.3
	0.2	14.7 x 14.7 x 14.7	14.7 x 14.7 x 14.5
	0.3	11.8 x 11.8 x 11.8	11.8 x 11.8 x 11.7
	0.4	10.4 x 10.4 x 10.4	10.5 x 10.4 x 10.3
	0.5	8.98 x 8.98 x 8.98	9.01 x 9.03 x 8.96
Kelvin Cell	0.15	11.7 x 11.7 x 11.7	11.8 x 11.7 x 11.7
	0.2	9.54 x 9.54 x 9.54	9.57 x 9.54 x 9.50
	0.3	8.40 x 8.40 x 8.40	8.44 x 8.43 x 8.39
Simple Cubic	0.1	10.5 x 10.5 x 10.5	10.6 x 10.5 x 10.4
	0.2	7.70 x 7.70 x 7.70	7.78 x 7.74 x 7.65
	0.3	5.90 x 5.90 x 5.90	5.96 x 5.95 x 5.86
	0.4	5.10 x 5.10 x 5.10	5.13 x 5.12 x 5.05
	0.5	4.10 x 4.10 x 4.10	4.32 x 4.33 x 4.24

Figure 4-8: CAD drawing and actual printed lattices of (a) & (d) Octet Truss lattice, (b) & (e) Kelvin Cell and (c) & (f) Simple Cubic structure, with $\rho/\rho_s = 0.3$.

The lattices were viewed under an optical microscope (OM) to analyse the printed strut shape and size of the six surfaces. Figure 4-9 (a) – (c) shows CAD design of a unit cell for the respective structures and (d) – (i) shows the optical images of the top and side faces of the Octet Truss, Kelvin Cell and Simple Cubic lattice.

The printed Octet Truss lattices' structure follows quite closely to the CAD design, with the average surface strut diameter of 0.47 ± 0.02 mm and the angle between the struts on the top and side faces differed by 1° . The top and bottom face of the Kelvin Cell was also close to the CAD design. The average surface strut diameter was 0.53 ± 0.02 mm. However, as pointed out with the red arrow in Figure 4-9 (h), the shape of the lattice holes on the sides (along the printing axis) differs from the actual design. Unlike the holes perpendicular to the printing axis where the circular hole is made up of a single polymerized layer, the holes along the printing axis are made up from many layers. In addition, the over polymerization required in the printing process as explained in Section 3.1.2 makes it difficult to accurately print a circular hole along the printing axis. To the best of the author's knowledge, there have not been any studies on the

effect of varying the lattice hole shape on the mechanical performance. Nevertheless, there are no sharp edges in these odd shapes to induce stress concentration points in the lattice. Hence, this should not have a significant impact on the mechanical performance of the lattice.

Figure 4-9: CAD design for (a) Octet Truss, (b) Kelvin Cell and (c) Simple Cubic lattice. Optical image of top and side faces of (d) & (g) Octet Truss, (e) & (h) Kelvin Cell and (f) & (i) Simple Cubic lattice.

The printed Simple Cubic structure had some differences from the CAD design. Figure 4-9 (f) shows the top side of the Simple Cubic lattice which had slightly rounded corners of the square hole (blue arrow). In addition, the edges at the joints were slightly jugged out which resulted in an uneven surface (red arrow). The average strut diameter for the top and bottom face was 0.45 ± 0.02 mm. On the other hand, shown in Figure 4-9 (i), the strut diameter also differed slightly across the Simple Cubic lattice; struts printed parallel to the printing axis (0.54 ± 0.01 mm) turned out to be thicker than those printed perpendicular to the printing axis (0.41 ± 0.03 mm). Different strut diameter can affect the mechanical performance of the lattice [16]. In light of this, the Simple Cubic lattices were tested in both directions.

Scanning Electron Microscopy (SEM) was used to look at the surface morphology of the lattices. Figure 4-10 (a) and (b) shows the SEM images of a broken piece from the top and bottom sides of a Kelvin Cell lattice, respectively, after compression test. As shown in Figure 4-10 (a), most struts are generally circular and have reasonably smooth surface morphologies with a few little bumps on the surface. However, the bottom layer of each structure has step-like surfaces as shown in Figure 4-10 (b). The average surface grain size of the lattice structure was 2.65 ± 0.2 μm , which is close to that measured from the cylindrical sample as shown in **Error! Reference source not found.** (2.39 ± 0.45 μm). This suggests that the different printed structures do not affect the sintering behavior.

Figure 4-10: SEM images of broken pieces of a Kelvin Cell lattice: (a) Top layer, (b) bottom layer and (c) strut surface of the lattice structure.

As mentioned in Chapter 3, the bottom of lattices must be thicker and have a larger contact with the base plate to provide stronger support for the whole lattice. To create this support, the base of the lattice was printed as a rectangle instead of a circle. Figure 4-11 shows a schematic to illustrate this concept. A circular strut as shown on the left has a smaller contact area at the base as compared to the rectangular base as shown on the right. The difference in this surface should have negligible effect on the mechanical properties of the structure as the steps are gradual, thus no stress concentration point should be created in the structure.

Figure 4-11: Schematic to illustrate contact area with surface of base plate when struts are printed in circular (left) and rectangular (right) shape.

4.2.1 Topology

Figure 4-12 shows the experimental relative modulus (E/E_s) and relative compressive strength (σ_c/σ_{cs}) against the relative densities (ρ/ρ_s) for Kelvin Cell, Octet Truss and Simple Cubic structures.

Figure 4-12: Logarithmic plot of (a) relative modulus (E/E_s) and (b) relative compressive strength (σ/σ_s) against relative density for Simple Cubic, Octet Truss and Kelvin Cell lattices.

From Figure 4-12, for any given relative density, the Simple Cubic structure clearly exhibits better stiffness and strength than both Kelvin Cell and Octet Truss. This is because Simple Cubic structures have 1/3 of their struts aligned with the loading axis while in the Kelvin Cell and Octet Truss lattices, all the struts are orientated at an angle to the loading axis. Aligned struts undergo ‘pure compression’ forces and are most effective in load bearing with the least amount of material. The structure was heavily reliant on these vertical struts for support against compression while the horizontal struts take the role of holding the vertical struts in place. On the other hand, the angled struts in Octet Truss and Kelvin Cell lattice would generate a moment under a uniaxial load and consequently, bending in the struts which induces tensile forces in the connecting joints. This is in addition to the tensile stress induced through holding the inclined struts together. Therefore, for the same strain, Octet Truss and Kelvin Cell will experience more tensile stress in the structure. Since ceramic is known to be much weaker in tensile than compression (i.e. 300 MPa vs. 2300 MPa for alumina), Kelvin Cell and Octet Truss are prone to premature failure in the presence of higher tensile stresses, thus leading to the lower relative strength as compared to Simple Cubic

lattices. However, it should be noted that although such slanted orientations give up some load bearing efficiency in a particular direction, they offer better isotropy in mechanical properties as the struts can bear similar load in different directions due to its orientation [17].

At a strain of 0.02%, the simulated loading conditions are seen in Figure 4-13. In Figure 4-13 (a), it can be observed that the compressive load was mainly supported by axial compression of vertically aligned struts in Simple Cubic lattices. Analysing just the tensile stresses in Simple Cubic lattice in Figure 4-13 (b), significant amounts of tensile stress occurred at horizontal struts near the joints, due to shear between the axially compressed vertical struts and the relatively unstrained horizontal struts. In other words, the vertical struts were indenting into the horizontal struts, resulting in surface bending and tensile stretching of the horizontal struts. Similar observation was made in the Octet Truss lattice but with about 3x higher tensile stress in the horizontal struts (Figure 4-13 (e)) and half the stress experienced on the diagonal struts (Figure 4-13 (b)) compared to Simple Cubic structure. Kelvin Cell lattices are designed to deform in a “bending-dominated” mechanism where the struts can rotate about the joints, resulting in stress concentration around regions with large moments. As a result, the lattice suffered from significant build-up of tensile stress at the joint regions (Figure 4-13 (f)), about 6x higher than in Simple Cubic structure, which led to failure at low nominal compressive stresses. The simulation outcomes align with the experimental results where the strength and stiffness of the lattices are the highest for Simple Cubic, followed by Octet Truss and lastly, Kelvin Cell (Figure 4-12).

Figure 4-13: (a) – (c) Von Mises stress distribution in Simple Cubic, Octet Truss and Kelvin Cell, respectively. (d) – (f) Maximum Principal (absolute) stress with only positive values being coloured highlighting regions with significant amount of tensile stress in (a) & (d) Simple Cubic, (b) & (e) Octet Truss and (c) & (f) Kelvin Cell lattices. Note that the stress colour scale varies across the images.

Following the Ashby analysis [18], the $E/E_s - \rho/\rho_s$ and $\sigma_c/\sigma_{cs} - \rho/\rho_s$ relationships in Figure 4-12 can be approximated by a power law (*e.g.* $E/E_s \propto (\rho/\rho_s)^P$). Table 4-3 summarizes the theoretical and experimental values of this relationship. There is a clear difference between the experimental and theoretical values of the exponents. This is because the theoretical values only consider cellular solids of low relative densities ($\rho/\rho_s < 0.1$) whereas in this study, a wider range of relative densities ($0.1 \leq \rho/\rho_s \leq 0.5$) are being investigated. As the relative density increases, the volume of joints and its contribution of the elastic properties will affect its performance and have to be accounted for. Experimental results have also revealed that this value can be subjected to changes as the relative density increases (Figure 4-12(b)).

Table 4-3: Exponent, P , of the power law relationship between relative stiffness and relative strength to relative density for the various structures.

	Domination	Simple Cubic	Octet Truss	Kelvin Cell
		Stretching	Stretching	Bending
Theory (Ashby)	Relative Stiffness	1.00	1.00	2.00
	Relative Strength	1.00	1.00	1.50
Experimental	Relative Stiffness	0.88	1.47	3.26
	Relative Strength	1.50	1.14	2.74

Simple Cubic lattice loaded perpendicular to the printing axis yielded significantly different results. Now that the thinner struts are vertically aligned with the loading axis and bear the compression load, the mechanical performance of the Simple Cubic lattices are weaker. Figure 4-12 (a) and (b) compares the relative stiffness and relative strength for Simple Cubic lattice loaded in perpendicular and parallel directions to the printing axis. When the structure was loaded perpendicularly, the mechanical properties were weaker. This is because the stress level on 1/3 of the vertically aligned “supporting” struts is now higher, thus, leading to lower strength.

In addition, when loading perpendicular to the printing axis, the top and bottom planes would fracture first before the whole lattice fails. Figure 4-14 (a) is an SEM image showing one of these bottom pieces that broke off first. These planes have juts at the sides which results in the lattice not being flat against the platen during the initial loading. Figure 4-14 (b) illustrates the forces acting on the lattice if the surface were to be flat. All the vertical struts would experience the same force and hence there would be minimal bending of the horizontal struts. On the other hand, Figure 4-14 (c) illustrates an uneven surface where the plane that is more protruding will bear more load. This induces a bending force and tensile stress on the horizontal struts connecting these planes. Due to ceramic being weak in tension, these struts will break off first during compression loading. This reinforces the importance of an even surface,

especially for ceramic lattices where the material performance changes significantly under different modes of forces.

Figure 4-14: (a) SEM image of a bottom plane that broke off first during compression loading. The dashed ovals highlight the uneven surface. Schematic illustration of the force intensity represented by the thickness of the red arrows on a Simple Cubic lattice with an (a) even surface and (b) uneven surface.

Compared to ductile lattices, the alumina lattices have better stiffness and compressive strength though at the expense of strain and thus energy absorption. Figure 4-15 shows that alumina Simple Cubic lattices have higher stiffness and compressive strength compared with Ti6Al4V Simple Cubic lattice [19]. At $\rho/\rho_s = 0.35$, the compressive strength of alumina Simple Cubic is 58.9% higher than that of Ti6Al4V Simple Cubic and 1400% stiffer. This demonstrated the potential of ceramic lattices to fill in the gap for lightweight-high strength and stiffness materials in applications such as aerospace.

Figure 4-15: Comparison of Young's modulus and compressive strength of alumina and Ti6Al4V [19] Simple Cubic lattice.

4.2.2 Relative Density

As seen in Figure 4-12, across the lattices, there is a direct relationship between the stiffness and strength to relative density. As the relative density increases, the difference between Kelvin Cell and Octet Truss shrinks such that the elastic mechanical properties are approximately the same for $\rho/\rho_s = 0.3$. This is consistent with results obtained previously by Oraib *et al.* [20]. In addition, the power law relationship of

relative compressive strength changes for Octet Truss at $\rho/\rho_s = 0.3$ as seen in Figure 4-12 (b).

Experimental data in Figure 4-16 was fitted with a modified Ashby relationship [18] in the simulation studies. The stretch-dominated lattices (i.e. Simple Cubic and Octet Truss) have the following equations:

$$\frac{E}{E_s} = a \left(\frac{\rho}{\rho_s} \right) + (1 - a) \left(\frac{\rho}{\rho_s} \right)^m \quad (\text{modulus, stretch-dominated}), \quad (4-2)$$

and

$$\frac{\sigma_c}{\sigma_{cs}} = a \left(\frac{\rho}{\rho_s} \right) + (1 - a) \left(\frac{\rho}{\rho_s} \right)^m \quad (\text{strength, stretch-dominated}), \quad (4-3)$$

while the bending-dominated lattice (i.e. Kelvin Cell) has the following equations:

$$\frac{E}{E_s} = a \left(\frac{\rho}{\rho_s} \right)^2 + (1 - a) \left(\frac{\rho}{\rho_s} \right)^m \quad (\text{modulus, bending-dominated}), \quad (4-4)$$

and

$$\frac{\sigma_c}{\sigma_{cs}} = a \left(\frac{\rho}{\rho_s} \right)^{3/2} + (1 - a) \left(\frac{\rho}{\rho_s} \right)^m \quad (\text{strength, bending-dominated}), \quad (4-5)$$

In these equations, $\frac{E}{E_s}$ and $\frac{\sigma_c}{\sigma_{cs}}$ represents the modulus and compressive strength normalized against the solid material (E_s and σ_{cs}) with a and m being the fitting parameters ($0 \leq a \leq 1$). In the case of a stretching dominated lattice, the first term “ $a \left(\frac{\rho}{\rho_s} \right)$ ” in Equation 4-2 is a linear law related to stretch-dominated behavior [17], where a is indicative of the loading efficiency at low relative densities. As for bending-dominated structures, the “ $a \left(\frac{\rho}{\rho_s} \right)^2$ ” term in Equation 4-4 corresponds to the widely-used parabolic law for modulus and the “ $a \left(\frac{\rho}{\rho_s} \right)^{3/2}$ ” term in Equation 4-5 refers to the fracture of struts due to bending moments. The second term “ $(1 - a) \left(\frac{\rho}{\rho_s} \right)^m$ ” in Equation 4-(2 – 5) is an empirical power law accounting for the increasing influence of joints at higher relative densities.

Figure 4-16: Experimental and numerical logarithmic plot of (a - c) relative stiffness and (d - f) relative compressive strength against relative density of Simple Cubic, Octet Truss and Kelvin Cell lattices. In the Simple Cubic plots, diamond symbols were tested in parallel while squared symbols were tested perpendicular to the printing axis.

Figure 4-16 shows the simulation data fitted with the semi-empirical models from Equations 4-2 to 4-5. At low relative densities, the lattices behaved as what was expected. The Octet Truss and Simple Cubic lattices followed a slope of 1 and Kelvin Cell followed a slope of 2 and 1.5 for its modulus and strength, respectively. The modulus and strength behavior are seen to be dominated by the effect of struts (1st term in Equation 4-(2 – 5), dotted lines) at low densities. As relative density increases, the modulus and strength behavior transitioned from ‘strut-dominated’ to ‘joint-dominated’ as the magnitude of the 2nd term (dashed lines) exceeded that of the 1st term. This resulted in a steeper increment gradient for the modulus and strength. However, this change is more pronounced in the strength of the lattices than the modulus. This is similar to what was observed for the experimental results. The transition point for the modulus of Kelvin Cell was not observable because the limited data adhered closely

with the parabolic law, resulting in Equation 4-4 being fitted with $a = 0$ that eliminated the 1st term entirely.

Overall, the values of a for compressive strength were much lower for ceramic lattices than ductile lattices where $a \approx 1/3$. This is because the lattices experience tensile stresses as a result of struts bending and stretching. Since ceramic is weaker in tension compared to ductile materials, ceramic lattices are prone to premature failure, leading to low relative strength of the lattice.

The above transition is also observable in the fracture pattern of the lattice as the relative density increases, predominantly in Octet Truss. Figure 4-17 shows the stress-strain curves for Octet Truss lattice at $\rho/\rho_s = 0.3$ and $\rho/\rho_s = 0.5$ and the still frames of the lattice structure corresponding to different points on the curve. Each fracture in the lattice was accompanied by a dip in the stress.

For Octet Truss lattices with $\rho/\rho_s \leq 0.3$, the unit cells fractured in stages as the lattice was being compressed. Figure 4-17 (aii) shows some of the areas in the lattice that broke off as pointed out by the red arrows. Despite this, the lattice was still able to bear some load as shown in Figure 4-17 (a) after the first steep drop at ‘ii’. A larger proportion of the lattice continued to fracture as shown in Figure 4-17 (aiii). This continued until the lattice eventually failed where bulk pieces of the lattice remain. The fractures were generally found to occur in a diagonal direction.

On the other hand, Octet Truss lattices with $\rho/\rho_s > 0.3$ have a different fracture pattern. The stress-strain curve is relatively smoother with fewer and less pronounced dips in the curve as compared to lower relative densities. This is likely due to relatively smaller pieces breaking off from the lattice, thus the lattice maintained bulk of its structural integrity and was able to continue bearing most of the load. There is also a short plateau region at the beginning of point ‘ii’ in Figure 4-17 (b), an indication of slight lattice structural deformation. At this stage, the load was being distributed throughout the lattice, therefore the structure will deform as a single entity. Thus, the load undertaken by the lattice did not increase as this is the phase where structural deformation of the lattice is occurring (i.e. strain increasing without stress increase). This deformation of 0.2% strain to the structure is too small to be captured by the high

speed camera, however, it is captured by the characteristic stress-strain curve plateau region. When the strain at joints exceeded the failure strain of alumina, the lattice shattered and failed catastrophically along an ‘X’ plane as seen in Figure 4-17 (biii). At fracture, chunks of the lattice burst outwards into small, majority strut-sized pieces, leaving two pyramid bulks at the top and bottom.

Figure 4-17: (a) Stress-strain curve of Octet Truss lattice ($\rho/\rho_s = 0.3$). (i) – (iv) Still frames from the high speed video capture of the compression testing. (b) Stress-strain curve of Octet Truss lattice ($\rho/\rho_s = 0.5$). (i) – (iv) Still frames from the high speed video capture of the compression testing. The loading direction is from top to bottom.

The change in the fracture as a result of relative density is due to the strut aspect ratio that determines a stable or unstable fracture mode. Mohr *et al.* [21] did a simulation study on the fracture response of stainless steel 316L Octet Truss lattices at different relative densities. Figure 4-18 (a) shows his simulation results for low relative density Octet Truss while Figure 4-18 (b) shows the high relative density Octet Truss simulation results. At low densities, the slender struts tend to form hinges at the centre which concentrates stress at that area and makes them prone to buckling. As such, they tend to have an unstable “twist-mode” fracture. These struts fracture in stages, starting the pseudoplastic regime shown in Figure 4-19. This causes shocks and vibrations in the remaining lattice, as reflected by the erratic nature of the stress-strain profile after failure (Figure 4-17 (a)). These shocks and vibrations can cause further damage to the lattice, hence, contributing to the step-like fracture. The pseudoplastic behaviour is an advantage over solid ceramic samples which fail catastrophically as this gives users time to respond before the specimen fails.

Figure 4-18: Simulation results of stainless steel 316L Octet Truss lattices density-dependent unit cell deformation mode: (a) twist mode at low relative densities and (b) stable mode at high relative densities [21].

On the contrary, due to the shorter node-to-node distance for higher relative density structures, the plastic hinges form at the nodes instead and start to interact with the neighbouring nodes. This creates a continuous zone of plastic deformation throughout the struts which leads to more effective load distribution and stable deformation. In turn, the pseudoplastic behaviour is reduced in high density structures. As a result, the lattices take on more external load which caused more build-up of strain energy in the structure as shown in Figure 4-19. Hence, when the lattice eventually broke at higher strains, this large amount of strain energy stored was released as kinetic energy that expelled the fractured struts with greater force and thus, further from its original position.

Figure 4-19: Typical stress-strain and energy-strain characteristics, taken from simulations of an Octet Truss lattice with 10% relative density.

Figure 4-20 shows the stress-strain curve of Kelvin Cell ($\rho/\rho_s = 0.15$) with the images of the broken lattice during the loading process. Similar to the low relative density Octet Truss lattices, Kelvin Cell exhibited pseudoplastic behaviour and fractures diagonally as seen in Figure 4-20 (iii) and (iv).

Figure 4-20: Stress-strain curve of alumina Kelvin Cell lattice ($\rho/\rho_s = 0.15$). (i) – (iv) Still frames from the high speed video capture of the compression testing. The loading direction is from top to bottom.

Figure 4-21 shows the stress-strain curve of Simple Cubic lattice ($\rho/\rho_s = 0.4$) with the images of broken lattice during the loading process. Similar to the high relative density Octet Truss, Simple Cubic lattices ($\rho/\rho_s = 0.2 - 0.4$) exhibited relatively lesser pseudoplastic behaviour than low relative density Kelvin Cell and Octet Truss. This is likely because Simple Cubic geometry do not have much tortuosity which allows load to be evenly distributed throughout the vertical struts. This enables the lattice to reach a higher stress and once the first horizontal strut fractures, the partially fractured lattice loses its stability and shortly after, fails completely, causing the effective stress to drop rapidly to zero.

Figure 4-21: Stress-strain curve of alumina Simple Cubic lattice ($\rho/\rho_s = 0.4$). (i) – (v) Still frames from the high speed video capture of the compression testing. The loading direction is from top to bottom.

Figure 4-22 shows the plot of failure strain and energy absorption at fracture in relation to the relative density for the different lattice structures. As shown in Figure 4-22 (a), the increase in failure strain is small and only seem to be more evident at higher relative densities. A possible explanation is that as the lattice is able to undergo higher stress, consequently, the lattice undertakes higher strain before it eventually fractures.

Figure 4-22 (b) shows the energy absorption up to failure for the three different lattices at various relative densities. The trend observed is rather similar to the strength and modulus of the lattice. With higher strength, modulus and failure strain, it is as expected that the energy absorption of Simple Cubic will be higher, followed by the Octet Truss and Kelvin Cell. The spread in values for alumina lattices could be due to the sensitivity of ceramics to defects. A minor defect in the lattice could result in a significant drop in the mechanical performance.

Figure 4-22: (a) Failure strain and (b) energy absorption up to fracture against relative density of Kelvin Cell, Octet Truss and Simple Cubic structure.

The fracture of lattices tends to occur at the joints of the lattices as observed in the still frames shown in the previous section (Figure 4-17, Figure 4-20 and Figure 4-21). This can be explained through the simulation work shown in Figure 4-13 (d) – (f) where there is built-up of tensile stress at the joints of the lattices.

Octet Truss (Figure 4-17) and Kelvin Cell (Figure 4-20) tend to fail along a diagonal plane while Simple Cubic (Figure 4-21) fractures along the vertical plane. At higher relative density, Octet Truss transits from a single diagonal plane to an ‘X’ plane

(Figure 4-17). This diagonal and ‘X’ plane is similar to the densification plane in metal Octet Truss and Kelvin Cell as shown in Figure 4-23 [20]. The same is observed for polymer lattices [22].

Figure 4-23: Made of steel, (a) Octet truss lattice and (b) Kelvin cell lattice after compression test [20].

Firstly, this suggests that the fracture pattern is dependent on the lattice geometry, regardless of the material and relative density. At the densification planes in the ductile Octet Truss and Kelvin Cell lattices, the struts were likely stretched as the struts did not buckle. This is similar to the tensile stresses observed in the simulation results for ceramic lattices. However, due to the brittle nature of ceramics, the alumina lattices fracture diagonally instead of densifying.

In contrast to Octet Truss and Kelvin Cell, Simple Cubic structures fractured vertically as seen in Figure 4-21 (ii) and (iii). This is due to the alignment of struts in the lattices. Simple Cubic structures have struts that are either perpendicular or horizontal to the loading axis. As such, load will be directed down the lattice, if not perpendicularly, following the strut alignment. On the other hand, Kelvin Cell and Octet Truss have struts that are aligned diagonally. This causes the load to be re-directed in a diagonal manner which led to the diagonal fractures of the lattices.

Through simulations, it was observed that the pyramidal-shaped fragments observed at the top and bottom of Octet Truss lattices at the end of the compression test were a direct result of boundary conditions imposed by the compressing plates. Figure 4-24 shows the simulation results for constrained and unconstrained lateral movement of the nodes during the compression test. Figure 4-24 (a) shows the post-failure of the

lattice when the surface of the lattice was being constrained laterally during compression. Most of the damage occurred at regions far away from the surface. The yellow dashed lines indicate the relatively undamaged region which resembles the pyramidal fragments observed in experiments (Figure 4-17 (biv)). When laterally constrained by the compression platens, the struts near the platens experienced very little strain or tension as shown in Figure 4-24 (a) and thus, very little damage. On the contrary, without the lateral constraints, the damage was more evenly distributed throughout the lattice (Figure 4-24 (b)). Damage also tend to start at the free edges along the lattice sides where there is no confinement (Figure 4-17 (bii) – (biii)), it then spreads towards the centre until the structure eventually collapses (Figure 4-17 (biv)), leaving behind the pyramidal fragment.

Figure 4-24: Comparison of damage (a) with and (b) without lateral constraints on the top surfaces of Octet Truss ($\rho/\rho_s = 0.5$). Image shows the post-failure and the 'X' at the bottom is the point of symmetry. (c) Comparison of the tensile stresses on the top surface when compressed to 0.02% effective strain. (d) Stress-strain curves of constrained and unconstrained lattices. All lattices were compressed along the vertical direction.

It is more difficult to observe this behaviour in lower density lattices as their slender struts break more easily due to shock vibrations in the pseudoplastic regimes (Figure 4-19). Therefore, the lattices only failed along one of the diagonal planes. Despite these confinements in the platens, Figure 4-24 (d) shows that it has no significant effect on the overall stress-strain characteristics.

4.2.3 Fracture Morphology

Figure 4-25 shows SEM images of fractured fragments and surfaces of Octet Truss and Simple Cubic lattices after compression test.

Figure 4-25: SEM images of (a) pyramidal fragment from Octet Truss ($\rho/\rho_s = 0.4$) after compression test and (b) a zoomed-in image of a crack. (c) & (e) Fractured surface of Simple Cubic ($\rho/\rho_s = 0.4$). (d) a zoomed-in image of grains showing inter-granular and trans-granular fracture. (f) a zoomed-in image of the pore and some agglomerated grains within the pore.

Figure 4-25 (a) shows one of the pyramidal fragments from an Octet Truss lattice ($\rho/\rho_s = 0.4$). It can be seen that the fractures occurred mostly at the joints as discussed in the earlier sections. Zooming in on a crack of this fragment (Figure 4-25 (b)), the crack is crooked. Comparing with the work done by Liu *et al.* [23], it appears that the Octet Truss exhibited an inter-granular crack where there were multiple crack deflections. Figure 4-26 (a) shows an example of an inter-granular fracture in ceramic which happens in a zig-zag pattern as compared to Figure 4-26 (b) showing a trans-granular fracture that is a relatively straight crack.

Figure 4-26: SEM image of ceramic with (a) inter-granular fracture and (b) trans-granular fracture [23].

Figure 4-25 (c) shows a fractured surface of a Simple Cubic structure ($\rho/\rho_s = 0.4$). Majority of the alumina lattice fracture surfaces have a mixed mode fracture consisting of trans-granular and inter-granular highlighted in Figure 4-25 (d). Such a fracture phenomenon is commonly seen in solid alumina made from conventional methods too [24], [25]. Figure 4-27 shows an SEM image of a fractured fine-grain alumina after bending test. It is commonly known that trans-granular is associated with brittle fracture and inter-granular with ductile fracture, implying the alumina lattices underwent brittle and ductile fracture concurrently.

Defect regions were also identified on the fractured surface. The defects were predominantly pores with agglomerated grains within as shown in Figure 4-25 (e) and (f). A region of agglomerated grains is also seen in Figure 4-25 (e) in the yellow dotted circle. Agglomeration has been a longstanding issue during the heat treatment process of ceramics as they weaken its mechanical properties [26]. Figure 4-27 shows an agglomeration in alumina with a crescent shaped pore surrounding it where the agglomeration looks somewhat similar to those found in alumina lattice (Figure 4-25 (f)). Such defect acts as failure initiation point in the structure and weaken their mechanical properties.

Figure 4-27: Fracture surface of a solid alumina sample after bending test [24].

4.3 Interpenetrating Phase Composites (IPCs)

Octet Truss lattices were infiltrated with epoxy to make epoxy-alumina Octet Truss interpenetrating phase composites (E-AOT). Due to difference in thermal coefficient of expansion (CTE), the epoxy matrix cracked when Octet Truss with relative density, $(\rho/\rho_s)_{\text{Lattice}} < 0.3$, was used. Thus, $(\rho/\rho_s)_{\text{Lattice}}$ ranging from 0.3 to 0.5 were fabricated. The following section will discuss the mechanical effects of infiltrating Octet Truss lattice with epoxy.

Figure 4-28 shows the compressive strength per unit weight and stiffness per unit weight against lattice relative density for Octet Truss lattice and E-AOT.

Figure 4-28: (a) Compressive strength and (b) Young's modulus per unit weight against relative density of Octet Truss lattice and epoxy-alumina Octet Truss interpenetrating phase composites (E-AOT).

As shown in Figure 4-28 (a), the compressive strength per unit weight of E-AOT increases with increasing lattice relative density. At $(\rho/\rho_s)_{\text{Lattice}} = 0.3$ and 0.4 , the normalized compressive strength of E-AOT is higher than the Octet Truss lattice. At $(\rho/\rho_s)_{\text{Lattice}} = 0.3$, the normalized compressive strength of E-AOT is 81.5% higher than its lattice form. At $(\rho/\rho_s)_{\text{Lattice}} = 0.4$, this improvement is 56.29%. This is because the epoxy matrix provides additional support to the structure. The epoxy acts as a confinement to hold the structure in place, preventing it from failing catastrophically as observed in the pure lattices in Section 4.2. It also helps in distributing the stress level to other parts of the lattice structure in the E-AOT. Instead of sudden failure like those

of the pure Octet Truss lattice, the failure of the E-AOT happens gradually and over a larger strain as will be elaborated in the subsequent section. As the lattice relative density increases, the normalized compressive strength of the two converges to meet at approximately $(\rho/\rho_s)_{\text{Lattice}} = 0.5$. This is possibly because the effect of the matrix in an IPC is dependent on the difference in Young's modulus between the reinforcement and matrix material [27]. As the Octet Truss relative density increases, so does its Young's modulus. This makes the difference in Young's modulus of the two materials even greater and hence, reducing the effect of epoxy as a matrix. Additionally, the amount of epoxy in $(\rho/\rho_s)_{\text{Lattice}} = 0.5$ is also lesser and thus may not be enough to provide sufficient support to the lattice as compared to lower density E-AOT.

Figure 4-28 (b) shows the Young's modulus per unit weight of E-AOT and Octet Truss lattice. As expected, the Young's modulus of E-AOT is in between the Octet Truss lattices (20 GPa at $\rho/\rho_s = 0.5$) and epoxy matrix (1.5 GPa), which is what is expected for a composite. However, unlike the compressive strength, the increase in Young's modulus (Figure 4-28 (b)) of E-AOT is relatively small, almost negligible. The Young's modulus per unit weight at $(\rho/\rho_s)_{\text{Lattice}} = 0.3$ is 1.5 GPa/g and at $(\rho/\rho_s)_{\text{Lattice}} = 0.4$ and 0.5 is 2.19 GPa/g. This is not the common trend observed in composites where the stiffness increases with higher volume percentage (*vol. %*) of the stiffer component. The large difference in the Young's modulus (200x) between both materials may have resulted in this outcome. However, further studies have to be done to verify the reason behind this.

Figure 4-29: Comparison of Young's modulus between this study and work by McGrath *et al.* [28] and Tilbrook *et al.* [29]

Figure 4-29 compares the Young's modulus of alumina-epoxy composites from this study against those from McGrath *et al.* [28] and Tilbrook *et al.* [29]. The results are comparable, in the same magnitude of order. Considering the fact the Young's modulus of Simple Cubic is at least 3 times of Octet Truss based on the results in Section 4.2, there is still much room for improvement in the IPC performance and potentially exceed conventional alumina-epoxy composites.

Figure 4-30: (a) Stress-strain curves of E-AOT with different $(\rho/\rho_s)_{\text{Lattice}}$. (b) Energy absorption (at strain = 0.1 and 0.15) against relative density of Octet Truss lattice and epoxy-alumina octet truss interpenetrating phase composites (E-AOT).

Figure 4-30 (a) shows the stress-strain curves of E-AOT at different relative densities. Overall, the strain is at least 100x higher than the standalone Octet Truss lattice. A common phenomenon is the initial peak stress of the samples before it plateaus at a lower stress. As the lattice relative density increases in E-AOT, the plateau duration after the peak stress gets shorter as well as the fracture strain. The decreasing strain with relatively minor increase in stress resulted in a decreasing trend in energy absorption (at strain = 0.1 and 0.15) as the $(\rho/\rho_s)_{\text{Lattice}}$ increases as shown in Figure 4-30 (b). It is likely that the stress was distributed to both components in the E-AOT

during the initial loading until the peak stress was reached. The subsequent loading process in the blue region in Figure 4-30 (a) is epoxy-dominated where the Octet Truss lattice would have likely cracked and the epoxy matrix was holding bulk of the load which corresponds to Figure 4-31 (b) – (d). This also explains why $(\rho/\rho_s)_{\text{Lattice}} = 0.3$ had a longer plateau region as it had more epoxy to take on further loading and strain, giving rise to higher energy absorption.

Figure 4-31 shows video images of the E-AOT at different strains. At the initial stage of loading up to the peak stress (Figure 4-31 (b)), there is slight buckling of the sample where the sides started bulging. It is likely that in this region, the lattice already exhibited some cracks but due to the epoxy matrix, the lattice was held together. As the loading proceeded, cracks started to become more pronounced and the sides also became more bent. Figure 4-31 (c) shows the E-AOT at the end of the plateau region whereby there were obvious cracks through the whole sample as well as in the lattice within the epoxy. The joints are seen to have broken off but still stuck in the epoxy matrix as pointed out by the yellow arrow. The first cracks usually start from the sides before moving to the centre, similar to the pattern of the lattice alone. However, other than some diagonal fractures observed, the E-AOT also fractured vertically which was not observed for the Octet Truss lattice. Past this stage, the sample lost its structural integrity and gradually crumbled, leading to broken pieces of various shapes and sizes. Some of the pieces still had the lattice within the epoxy but there were also a handful where the alumina lattice and epoxy broke off as two individual components. This suggests that there is weak interfacial bonding between the two phases. In addition, the sides were bent as traced by the yellow dash in Figure 4-31 (d).

Figure 4-31: Still frame images from high speed video of E-AOT with $(\rho/\rho_s)_{\text{Lattice}} = 0.5$. (a) $\varepsilon = 0$, (b) $\varepsilon = 0.03$, peak stress, E-AOT showing slight bulge at the sides. (c) $\varepsilon = 0.08$, end of plateau region. (d) $\varepsilon = \varepsilon_f$ (0.16), failure.

Figure 4-32 (a) shows the SEM image of a fractured strut from the IPC. Generally, epoxy pieces were not seen left on the struts of the lattice other than at the joint corners. Figure 4-32 (b) shows the interface between the alumina strut and epoxy matrix. There is a gap between the alumina strut and epoxy fragment left behind. Figure 4-32 (c) shows the surface of the strut as boxed up in blue in Figure 4-32 (a). Although some epoxy bits can be seen on the surfaces, there are no big chunks seen on the struts away from the joint area. This indicates that there was none or if present, weak bonding between alumina and epoxy. As such, the presence of epoxy solely provides structural support without any chemical bond to aid its mechanical performance.

Figure 4-32: E-AOT SEM image of (a) a broken strut, (b) interface between epoxy and alumina and (c) strut surface showing some islands of epoxy fragments on alumina.

4.4 Conclusion

In conclusion, due to the brittle nature of ceramics, ceramic lattices perform differently from what is commonly observed in ductile lattices. First and foremost, there is minimal densification and strut bending due to the brittleness of ceramic. Thus, ceramic lattices do not perform as well when it comes to structures that have bending and stretching as their dominant fracture mode because it induces tensile stresses in the supporting struts. That being said, the experimental results have proven that when a structure is supported by struts that undergo compression during loading (e.g. Simple Cubic structure), the lattice yields a much better mechanical performance than similar lattices made of ductile materials. For the same relative density, the alumina Simple Cubic in this study is 58.9% stronger in compression and 1400% stiffer than a Ti6Al4V Simple Cubic structure [19]. Therefore, there is potential for ceramic 3D printing to push the boundaries of structural lattices which was once only available in polymers and metals. Knowledge on fracture behaviour of ceramic lattices from this study can be utilized in designing mechanically stronger lattices.

Adding epoxy to alumina Octet Truss lattice did improve the compressive strength, strain and energy absorption compared to the pure lattice itself. Compared to conventional composites, the stiffness of E-AOT with epoxy-alumina composite are comparable [28], [29], this could suggest that Octet Truss may not be a suitable structure for ceramic IPCs. The above studies on lattice structures have demonstrated the sensitivity of ceramic to geometry where Simple Cubic performed significantly better than Octet Truss and Kelvin Cell.

In essence, this study demonstrated the possibility of 3D printing ceramic lattices and its potential in the lattice material group due to its stronger and stiffer properties. However, it should be noted that due to the nature of ceramics, it is very sensitive to defects, hence, the printing process has to be very well controlled.

4.5 References

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Chapter 5

Conclusions and Recommendations

This chapter will summarize what has been done in this study and its scientific implications. The future work on improving the mechanical properties of ceramic lattices and interpenetrating phase composites will also be discussed.

5.1 Conclusions

In summary, the mechanical performance of ceramic lattices and interpenetrating phase composites have been studied. Through a combination of computer simulation and experiments, it was found that the type of failure observed for ceramic lattices is very different from ductile materials such as polymers and metals. Due to the brittleness of ceramics, the struts experience brittle failure before densification can happen. Amid its brittleness, ceramic lattices exhibit gradual fracture (pseudoplastic behavior) where it shows some initial fracture before catastrophic failure. Such a behavior is not seen in conventional solid ceramics where they fail without any warning.

Studies were done to understand the influence of relative density and topology on the mechanical properties of ceramic lattices. The Simple Cubic structure had 1/3 of its struts aligned to the loading axis where they undergo compressive forces during loading. On the contrary, Kelvin Cell and Octet Truss lattice had their struts angled to the loading axis which generates bending in the struts and induces tensile stress. This difference in strut alignment and forces resulted in Simple Cubic lattice performing at least 10x stronger and 5x stiffer than the other lattices of the same relative density. Increasing the relative density of the lattices improves its compressive strength, stiffness, failure strain and energy absorption ability. Simulation results have shown that as the relative density increases, the fracture determining factor shifts from predominantly struts-limited to predominantly joints-limited. Piecing the above information together, the greater the volume joints occupy, the more stable the fracture is and thus the better the lattice performance. The fracture of 3D printed ceramics is no different from what is commonly seen in alumina fabricated through conventional methods. The lattices exhibit a combination of inter-granular and trans-granular fracture and still possess the issue of agglomeration which occurs during the heat treatment process.

A method to fabricate interpenetrating phase composites was established in this study. With epoxy as the matrix material, the epoxy-alumina Octet Truss interpenetrating phase composite (E-AOT) showed improvement in their compressive strength, strain and energy absorption compared to the standalone alumina Octet Truss.

However, improvements can be made given that Octet Truss is not the best performing lattice when it comes to ceramic material. In addition, the large difference in Young's modulus and weak interfacial bonding between alumina and epoxy matrix also limits the strengthening effect of the matrix in E-AOT. Preliminary studies using Simple Cubic lattice in the IPC have shown promising improvement in the mechanical properties.

Figure 5-1 shows the Ashby plot of Young's modulus against density. Mapping the results in this study to what is currently available, Simple Cubic structures with $\rho/\rho_s = 0.3$ and $\rho/\rho_s = 0.4$ have stiffness outside the range of what is currently available. With density in the polymeric range, they have stiffness in the range of 30 – 45 GPa which is similar to that of metals and composites; this is about 10x stiffer than polymers. In addition, the lattices with $\rho/\rho_s = 0.1$ and $\rho/\rho_s = 0.2$ lie close to the edge of the bubble of natural materials. These lattices still have relatively good stiffness and do fill the small gap between natural materials and non-technical ceramics.

Figure 5-1: Ashby chart plotting Young's modulus against density. The Young's modulus of alumina Kelvin Cell, Octet Truss, Simple Cubic and E-AOT IPC has been marked out on the chart.

Figure 5-2 shows the Ashby plot of compressive strength against density. Similarly, of particular interest are the Simple Cubic lattices. Having the density in the polymeric range ($\rho/\rho_s = 0.3$ and $\rho/\rho_s = 0.4$), these lattice samples were able to perform as well as the upper percentile of metals and composites. Lower density Simple Cubic lattices ($\rho/\rho_s = 0.1$ and $\rho/\rho_s = 0.2$) also perform at the upper percentile of natural materials.

Figure 5-2: Ashby chart plotting compressive strength against density. The compressive strength of alumina Kelvin Cell, Octet Truss, Simple Cubic and E-AOT IPC has been marked out on the chart.

Therefore, combining the experimental findings from the studies conducted above, the design of the lattice can be modified to improve its properties. Some modifications to the Simple Cubic lattice could potentially fill the empty space above polymers where it will possess the density of polymers while having the compressive strength of stronger metals.

5.1.1 Scientific Significance

Ceramic lattices combine the advantage of material properties with geometrical strength to give a stronger lightweight material. This study has proven the feasibility of 3D printing of ceramics which has been a technology more commonly available to polymers and metals. Being able to print stiffer and stronger materials widens the applications for 3D printed lattices. Compared with metals and polymers, ceramics can withstand higher temperature operations and other harsher environments. For example, armor protection can tap on the higher stiffness and strength per unit weight of ceramic lattices. In the medical field, body parts specific to individuals can also be printed to tune the properties of the ceramic part (e.g. parts modulus and strength), to improve patient's comfort and lifespan of using an implant due to ceramic being more wear resistant.

The studies on alumina lattices have provided valuable insights to how ceramics will perform as a lattice. With a deeper understanding to the way they behave, it lays the groundwork for designing ceramic lattices. As ceramic is very brittle, bending and tensile stresses within the lattice have to be minimal in order for the ceramic lattice to perform well. This fracture mechanism is very different from ductile materials like polymers and metals which has been widely studied. On top of that, results from this study allows scientist to incorporate the experimental results into their simulations to verify the numerical calculations and validate further simulation studies.

The study on IPCs proves the potential of a second phase to improve the performance of lattice structures. In addition, this study lay down the groundwork for future IPC study by providing an initial framework with suitable structure for ceramic IPC study and fabrication process. On top of that, this study sheds light onto the

structure performance sensitivity of the sample to its printing orientation. This would help researchers that are working on IPC and ceramic lattices samples in future.

5.2 Future Recommendations

Based on the current results obtained, more experiments can be done to improve the ceramic lattice system and gain a better understanding of ceramic lattices as well as the IPC behavior. The following sections will elaborate on future work in continuation of the current study.

5.2.1 Ceramic Lattices

Through understanding the reason behind the better performance of Simple Cubic lattices and testing the lattice in two different directions (i.e. perpendicular and parallel to the printing axis), more adjustments can be made to the design to improve its mechanical properties. The studies have shown that when the supporting struts are thicker, the lattice is able to withstand greater compressive stress. Therefore, Simple Cubic lattices can be designed to have the struts parallel to the loading axis thicker. From the current results, such a modification could improve the compressive strength and stiffness of the Simple Cubic structure.

With alumina being so brittle, other ceramic materials could also be experimented to understand how a slightly more ductile ceramic material changes the mechanical properties and fracture mechanism. In recent testing, Octet Truss lattices were printed using zirconia. Zirconia is less stiff (42% lower) but has a higher flexural strength (172% higher) compared to alumina. Compared with alumina, the compressive strength of zirconia Octet Truss at $\rho/\rho_s = 0.5$ was 70% stronger with approximately the same stiffness (i.e. the average Young's modulus of alumina Octet Truss was 20 GPa and that of zirconia Octet Truss was 23 GPa). However, the density of zirconia is higher which brought the difference in mechanical performance down when normalized by density. This study has shown that a material of high flexural strength could yield promising mechanical performance. With a deeper understanding of the mechanism when the intrinsic material property changes, materials used can be optimized to improve the properties of ceramic lattices.

Subsequent studies may also include triply periodic minimal surfaces (TPMS) structures. These geometries are mathematically-known to minimize the effects of stress concentrations and have superior mechanical properties [1]. It would be valuable to understand if this calculation benefits brittle lattices as well.

5.2.2 Interpenetrating Phase Composites (IPC)

IPC experiments in this study have shown that adding a second phase does improve the compressive strength, strain and energy absorption of the lattice though at the expense of the Young's modulus. Surpassing the performance of E-AOT, preliminary studies on epoxy-alumina Simple Cubic IPC (E-ASC) showed a significant improvement in the compressive strength (325%), Young's modulus (487%) and energy absorption (135%) at $(\rho/\rho_s)_{\text{Lattice}} = 0.5$ as compared to E-AOT at the same lattice relative density. Therefore, future studies should continue to work on Simple Cubic structure IPCs as these preliminary results have revealed the potential of ceramic IPC when the correct geometry is used.

Previous studies reported that a large difference in the Young's modulus of both components would hinder the stress transfer from the matrix to the stiffer lattice within the IPC, thus deeming the system ineffective [2]. Future experiments could use a stiffer matrix material closer to alumina. For example, experimenting with a matrix material with closer Young's modulus to alumina, such as aluminium or steel (i.e. 69 GPa and 200 GPa). With closer Young's modulus and higher tensile strength (100 MPa - 500 MPa), aluminium will be able to provide a much better confinement to the ceramic lattice and stress distribution within the IPC. On top of that, in order to infiltrate ceramic lattice with aluminium, a high temperature process will be required to melt the aluminium before using technique such as squeeze casting or gas pressured infiltration. This coupled with the large CTE difference between aluminium and alumina, the aluminium matrix may provide a large prestress on alumina lattice, thus creating net compressive stress within the lattice during loading to improve its mechanical properties. In addition, based on the results of lattice geometries and initial experiments, Simple Cubic structures was shown to yield better performance than the Octet Truss. Hence, IPC using Simple Cubic structures may be a good starting point for future studies.

On top of that, the current study focused on the quasi-static mechanical properties. One recommendation is to study the dynamic properties of the IPC using Split Hopkinson Pressure Bar to understand the properties of IPC under high speed loading condition. The IPC will have high temperature potential if the matrix material is changed to metallic materials. During the study of metal/ceramic IPC, it is recommended to understand the thermomechanical properties of IPC using high temperature quasi-static compression test.

Interfacial bonding between matrix and reinforcement phase in composite had been shown to be critical for good composite performance [3], [4]. In a study done by Ji *et al.*, they found that nano-alumina particles grafted with polystyrene and polyacrylamide did improve the interfacial bonding between the epoxy matrix and alumina particles [5]. Therefore, study on improving the two components of IPC should be carried out to bring out the full potential of ceramic-based IPC. With the presence of strong chemical bonding between both components, two separate materials can potentially become a stronger, and perform similar to a monolithic material.

More studies can be conducted to analyze different combination of materials and structures. With that, both components can be mixed and matched to find suitable IPC properties for various types of applications. This brings out the advantage of 3D printing and IPC where it is a highly controllable system allowing specific changes to meet the needs of the users.

5.3 References

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