

HEAT TRANSFER OF 316L – A356 INTERPENETRATING HYBRID MATERIALS

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ABSTRACT

Rising global temperatures and more frequent extreme weather events as a result of climate change have caused countries to tighten sources of emissions from all sectors, most notably in transportation. Regulations, in the form of fuel economy mandates and removal of internal combustion engine vehicles from city centers, have forced automotive companies to increase the efficiency of the powertrains in the products they sell to consumers. In seeking higher efficiencies – whether for internal combustion engines or in fully electrified powertrains – automotive companies are demanding more from the materials they use. Alloying, as in other industries, has been relied on to deliver new levels of performance; however, the incremental improvements from alloying often trail the rate of technological progress desired.

Hybrid materials – a class of materials that are a combination of 2 or more monolithic materials to form one single “material” – offer an opportunity to hasten the time to achieve a collection of properties desired. In combustion applications, mechanical performance and heat transfer are critical to the overall powertrain efficiency. Despite the knowledge of hybrid materials, there exist few tools to understand the heat transfer within hybrid materials in applications where mechanical performance and heat transfer are critical.

The study aims to show the heat transfer of an interpenetrating hybrid material using an experiment developed for monolithic materials joined with a thermal simulation to understand where 1-dimensional assumptions break down. The heat transfer within the hybrid material will reveal a complex interaction of the bonding between the material combinations and their geometry:

1. Initial work developed a two-step process to produce metal-metal hybrid materials of varying volume fractions of 316L stainless steel and A356 – an aluminum-silicon alloy. The hybrid materials were characterized for compressive, tensile, thermal conductivity and diffusivity, and porosity.
2. The final work used the flash disk method to calculate the apparent thermal diffusivity through the samples. These values were then compared to a heat transfer finite element model of the different volume fraction hybrids. The results were then compared to the effects of the porosity within the samples, contact conductance, and breakdown of 1D heat transfer assumptions.

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Chapter One

INTRODUCTION

Motivation

Human accelerated climate change threatens the quality of life of the global population as nations face more frequent and more devastating extreme weather events and sea level rise. Much of the acceleration in global temperature since the Industrial Revolution can be traced to the period's rising consumption of energy from fossil fuel sources that release high global warming potential emissions into the atmosphere. In response, several climate agreements have directed their attention to reducing global emissions with promises put forward by each signing country. One of the largest single sources of carbon emissions is from the transportation industry. Since the first oil embargo of the 1970s, then for the purpose of reducing oil imports that were in short supply, Europe and the United States have set fuel efficiency standards – later as carbon emission standards – and emission standards for vehicles sold in their respective markets. As the dire consequences of human-accelerated climate have become more visible and better understood, further tightening of fuel efficiency standards has occurred.

While many cities and countries have begun to announce internal combustion engine bans in 2030, 2040, 2050, electric vehicle penetration remains limited due to a variety of technical and market challenges. To meet upcoming standards, vehicle manufacturers are looking to improve the engine efficiency with new combustion strategies, materials, and engine architectures. One method that has been used has been to add transmission gears (down-speeding) and downsize the engine to improve efficiency often paired with a turbocharger to match the power requirements previously held with a larger engine. [1] Nearly 60% of new vehicle sales in the United States features a combination of 6 or more speeds with a turbocharged gasoline engine. This combination places the operating range of the engine closer to the optimum brake thermal efficiency of the engine; however, this location is also near an unstable combustion region known as low speed pre-ignition (LSPI) or “superknock”. [2] When operating in LSPI conditions, a semi-stochastic process whose origin is still an active area of research [3–6], one cycle out of a million can cause a large pressure shock wave at the wrong time in the combustion process, as seen in Figure 1, causing permanent deformation to the piston. Because one cycle out of a million can translate into 10 minutes in an engine running during a normal operation, manufacturers avoid operating near the conditions that cause LSPI [7] avoiding

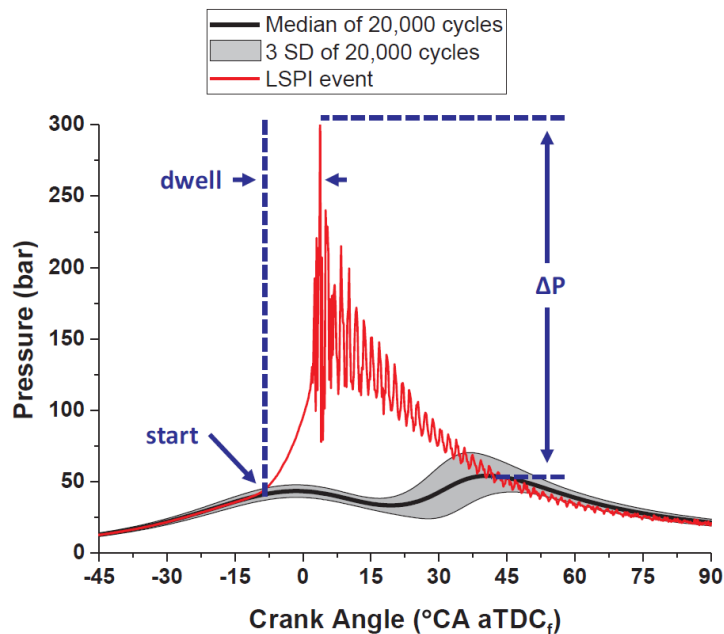


Figure 1 Superknock - LSPI - event causes a large pressure rise right as the piston is near at top dead center (crank angle of 0) that is much higher than the normal rise in pressure after the piston passes top dead center and even higher than in events when knock occurs. From Jatana, et al. [3]

LSPI; however, manufacturers also forgo small efficiency gains that could otherwise be achieved.

When a pressure wave travels down in an LSPI event, the small gas space between the piston and the cylinder wall concentrates the wave before striking the piston surface, often at the first or second ring land indentation in the piston near the oil cooling hole as seen in Figure 2. At this weakest point of the piston, the stress-induced shock loading exceeds the ultimate tensile strength of the Al alloy, causing a fast fracture. [8] In other high loading piston applications – heavy duty compression-ignition engines – the tradeoffs in piston weight and ultimate strength have seen piston manufactured with a clad ring surrounding the piston of a higher strength material. For light-duty downsized engines, the additional weight of this arrangement brings disadvantages to efficiency that outweigh the gains of operating in an otherwise higher efficiency range. The placement of a higher strength material locally within a piston region requires a new manufacturing process. How a material should be placed, and with what geometry requires new design rules that are not currently available. An engine piston is an example of a highly complex engineering challenge that must account for a combination of high



Figure 2 A damaged ring carrier piston following a severe LSPI event in a downsized turbocharged spark-ignited engine that fractures the second ringland. From Passow et al. [8]

temperature, zero creep, elastic deformation, surface resistance to combustion and combustion products, high stress rate, and high strain rate. There exist no complete set of design rules to create hybrid materials for mechanical and thermal properties, this thesis aims to address the complexities associated with thermal diffusion in hybrid materials, with preliminary work shown on compressive and tensile properties.

Fundamentals of Mechanical and Thermal Characterization

Characterization of Mechanical Properties

A standard measure of a material's performance in loading often starts with a uniaxial loading test, both in tension and in compression. The ASTM E8 [9] and ASTM E9 [10] standards provide guidance for testing metallic materials in tension and compression, respectively. For metals, compressive performance and tensile performance often mirror one another in elastic deformation (that is, the elastic Young's Modulus in compression is often the same as in tension), making it advantageous to study in compression. The standards are general in purpose – they do not restrict their applicability to monolithic materials – and have been used to test a variety of metallic hybrid materials [11–14]. In a uniaxial test, load is applied and a displacement is recorded in real-time. In measuring the initial area of the surface perpendicular to the uniaxial loading direction, as well as the initial length along the loading direction, engineering stress and strain can be calculated to produce a stress-strain curve. However, this curve is a global snapshot of the material's stress state in response to a strain, which for monolithic materials is mostly valid in the elastic region, which is most often not the case in hybrid

materials. Techniques like digital image correlation (DIC) can be used to reveal the stress states between the different constituents within the hybrid material tested. DIC is a pixel tracking technique that can relate the changes in pixel locations (displacements) to local strain fields. [15,16] The strain fields reveal areas of localized deformation where the resultant stress may be much higher than in the global properties (even beyond the point of yielding even if the global hybrid material appears to remain in elastic deformation).

Characterization of Heat Transfer

Thermal diffusivity (α) is the controlling property in transient conduction, where it measures the ability of a material to conduct heat relative to its ability to store thermal energy [17]. In a sense, thermal diffusivity is a measure of thermal inertia: materials with high thermal diffusivity change temperature quickly, while materials with low thermal diffusivity change temperature slowly in response to an external rise in temperature. [17] Thermal diffusivity can be measured with a single-flash disk method. [18,19] In this method, a short pulse of a defined heat flux from a high intensity lamp heats one surface and the rise in temperature is recorded on the opposite surface. This rise in temperature recorded is fit to a curve first devised by Parker [20], where an error minimization routine, such as Nelder-Mead simplex algorithm [21], can be used to find the characteristic half rise time in temperature that then can inform the calculated diffusivity, using equation 1 below. In equation 1, L is the thickness of the sample, α is the thermal diffusivity to be solved for, t is the time at which the temperature, T , is evaluated, with 8 terms (n) of the summation expansion used to balance precision and computational time.

$$\frac{T(t) - T_{min}}{T_{max}} = 1 + 2 \sum_{n=1}^{\infty} (-1)^n (-n^2 \omega) \quad (1)$$

$$\text{where } \omega = \frac{\pi^2 \alpha t}{L^2}$$

While the rise in temperature is often measured with a thermocouple for a global diffusivity, infrared thermal imaging cameras have also been used to create a pixel-level map of thermal diffusivity measurements, as equation 1 can be applied at the pixel level as recorded by the infrared camera over the short duration of the pulse. The applicability of this method, however, originally extends to only homogenous materials. It has been used in several studies of hybrid materials [22–24] to provide global properties of the hybrid materials. To date, there have been few studies that have studied the spatial distribution of thermal diffusivity measurements. [25]

Chapter Two

LITERATURE REVIEW AND SCIENTIFIC GAPS

Literature Review

Rules for Hybrid Materials

Hybrid materials as defined by Ashby [26], combine the properties of two (or more) monolithic materials, or of one material and space. Hybrid materials offer the opportunity to explore combinations in material properties that are possible but difficult (and/or expensive) [27] to achieve with conventional material property enhancements through alloying, polymer chemistry and other processes. In material selection, property charts can be produced to reveal advantageous materials for a given target mix of properties. In hybrid materials, property charts can inform material combinations to first investigate to deliver the range of properties desired. Hybrids can be characterized in four different arrangements [28]: cellular structures and foams, fibrous and particulate composites, sandwiches, and natural materials.

Cellular Structures and Foams

Cellular structures and foams are most often a combination of a single monolithic material and a gas (often air), whose gas properties may prove negligible to the material's overall mechanical properties, but otherwise prove desirable in conductivity, dielectric constant, and other physical properties. [29] Foams are most often monolithic materials that through different processes can become expanded with a foaming agent. Foam and cellular structures can be further subdivided into two general classifications based on their mechanical response to loading: bending-dominated and stretch-dominated. Bending-dominated [30] structures flex at the nodes of the lattice in response to mechanical deformation; foams typically are bending-dominated structures. Stretch-dominated structures, however, can see both compressive and tensile stresses across the struts, leading to the struts to “stretch” in deformation. It has been found that in situations where stiffness is of greatest concern, a stretching-dominated lattice can have up to ten times the stiffness of a bending-dominated configuration of the same material. Bending-dominated configuration, however, can find use in applications where energy absorption is critical [30].

Hierarchical materials, a term used throughout this study, also known as lattice materials [31], more specifically refer to cellular structures with a very

controlled order and hierarchy [32], These structures are considered “materials” only if the wavelength of loading is much larger than the collection of elements [31]. Hierarchical materials are generally a repeated unit cell of uniform sub-elements, typically beams, oriented in a stable configuration, often along the edges of polyhedral. The properties of these materials are dictated by the connectivity of the nodes and the volume fraction of the material that forms the repeating set of beams. [30] A bending-dominated configuration of a hierarchical material – where the matrix material is air – have been found to have a young’s modulus (E) that scales to the square of the relative density (ρ) of the beam material (b) and an initial yield stress (σ_y) that scales to 3/2 power of the relative density of the beam material (b) [31]:

$$E \sim \bar{\rho}_b^2 E_b \quad (2)$$

$$\sigma_y \sim \sqrt{0.3} \bar{\rho}_b^{3/2} \sigma_{y,b} \quad (3)$$

A stretch-dominated lattice, on the other hand, has a Young's Modulus and initial yield strength that vary linearly with density, bringing the material closest to the upper limit of the Hashin-Shtrikman bounds for a multiphase material [33]:

$$E \sim 0.3 \bar{\rho}_b E_{y,b} \quad (4)$$

$$\sigma_y \sim \sqrt{0.3} \bar{\rho}_b \sigma_{y,b} \quad (5)$$

Interpenetrating phase composites

Interpenetrating phase composites (IPCs) are a version of cellular structures and foams where two or more solid phases are contiguous throughout the material, leaving no phase completely isolated [34,35]. In most traditional manufacturing methods, the ability to make a hybrid of two or more contiguous phases is difficult if not physically impossible; however, several methods have been used previously to manufacture IPCs.

Manufacturing Hybrid Materials

Extrusion 3D Printing

In non-metallic composites, fiber-filled epoxy-based inks can be used to create fiber-aligned cellular structures [36]. Compton and Lewis added SiC whiskers and chopped carbon fibers to an epoxy-based ink consisting of Epon 826 epoxy resin, nano-clay platelets, and dimethyl methyl phosphonate (DMMP). The nano-clay platelets (1 nm thick; 100 nm in length) primarily served as a rheology modifier that imparted both shear thinning behavior and a shear yield stress to the uncured ink, while DMMP served to reduce the initial viscosity of the resin to allow for more fibers and whiskers to be added while still able to flow. [36] The high

aspect ratio fibers aligned under the shear and extensional flow field that developed within the micronozzle during printing that resulted in enhanced stiffening in the cured composite along the printing direction. [36] With this capability, the spatial orientation of the fibers can be controlled during the build process, further optimizing the characteristics of the cellular configurations designed. This control can happen at each individual build path, allowing for different orientations of fiber to be used within the same layer. This could allow for the development of functionally graded materials (FGMs) for engineered gradual transition in microstructure and/or composition to correspond to a desired change in properties along a desired direction [37] by varying the orientation and amount of reinforcement in the ink at time of deposition. Extrusion 3D printing holds great promise particulate reinforcement within cellular materials using thermoplastics. The next sections will discuss hybrid materials of two or more solid phases, starting first with a sandwich structure in ultrasonic additive manufacturing (UAM).

Ultrasonic Additive Manufacturing

Combinations of metallic sheets when fused together make a sandwich hybrid material where the total properties across the sandwich are a combination of the layers. One way to introduce multi-materials to solid fabrication is through ultrasonic additive manufacturing (UAM) – where a sonotrode is placed on a new strip layer on top of existing layers and then localizes the deformation in an ultrasonic pulse to deform the new sheet to the existing part. [38] The heat and stress applied sonotrode-induced friction between two strips causes severe plastic deformation between the two layers as the oxide layers between the asperities of each surface rupture, allowing metal-to-metal contact that eventually bonds in dynamic shearing. [39] This process has a great advantage versus powder bed additive manufacturing processes in that it can occur at room temperature in an open environment. At this time, UAM, is limited in the geometry of parts that it can produce, but as mentioned can be use in gradient applications, in both metal-metal composites, as well as the inclusion of sensors and other features that would be difficult to embed otherwise. [25]

Indirect Fused Deposition

Indirect fused deposition [40–42] is a multi-step process – that is similar to lost-wax casting – that first uses fused filament fabrication (FFF) – a layer by layer thermoplastic filament melting process – to deposit a sacrificial polymer that is the negative of the planned metallic reinforcing phase. A slurry of ceramic powder, alcohol, and binder infiltrates the polymer and is then dried. Following the drying of the ceramic slurry, the ceramic preform is first heated to an elevated

temperature to remove the binder before being raised to an even higher temperature to sinter the ceramic to densify. Finally, the sintered ceramic preforms are melt-infiltrated with a metal of choice. This process can be used to make set volume fractions of metallic ceramic IPCs or functionally gradient IPCs. [41] The advantage of the two-step process is that the separation of the formation for each of the constituents avoids undesired mixing of the two (or more) constituent materials that could lead to intermetallic formation, many of which can lead to brittle cracking during the build process.

Binder-Jet Infiltration

Applying the concept of print then infiltrate, binder-jet infiltration is a two-step open air process that requires the use of a furnace without or without subsequent melt infiltration to produce a dense additively produced part. [38] Using a powder bed, a water-based binder is deposited much like in a desktop inkjet printer in the pattern corresponding to the specific slice desired. The subsequent “green” part produced must carefully be removed before being sintered in a kiln. Because the part can still be only 60% dense (due to packing factor of the original powder) even following the initial sintering, melt infiltration of a similar (or dissimilar) material is necessary to lower the porosity to 5% or less. While often the melt infiltrant is another metal, resins and thermopolymers can also be used where direct mechanical performance is not important as in the case of near-net-shaped permanent magnets developed by Paranthaman et al. [43].

Direct Additive Manufacturing of IPCs

More recently, there have been several attempts to produce metal IPCs with direct additive processes (unlike the indirect fused deposition). Martin and co-workers [44] developed a two-step additive process to produce an IPC with Ti-6Al-4V as the reinforcing phase with commercially pure Ti Grade 2 as the matrix phase, as well as show the capability with the same alloy: two powder sources of Ti-6Al-4V of different oxygen content. Electron beam melting (EBM) creates a near fully dense (0.4% porosity) lattice structure of Ti-6Al-4V and is then placed into a die of the powder for the matrix. EBM is a powder-based additive process that applies an electron beam to melt powder at each layer to produce a dense part in a controlled high vacuum environment [38] Spark Plasma Sintering (SPS), also known as pulsed electric current sintering (PECs), is then used to sinter the loose powder into a fully consolidated phase. SPS is an additive process – also under vacuum – that combines heat and pressure as in conventional sintering to sinter loose particles to their near theoretical density, based on the powder’s packing factor. The heat source is not applied by external heating devices, rather a pulsed

DC current is passed through the die and conductive powder causing joule heating to supply the temperature necessary locally within the loose powder to promote sintering. [45] This technique produced near fully dense parts with pores spread randomly throughout and not preferentially at the boundary between the EBM-produced reinforcement and SPS-produced matrix. While this technique holds promise to manufacture metallic IPCs, it has not been extended to mixed material systems where wetting may be of concern.

Topology Optimization

With the usefulness of hybrid materials well understood, computational resources to understand how to use multi-materials or existing materials better have been the focus of ongoing research effort since the 1990s, starting with truss structures, [46–49], with the original theory recently rediscovered [38] from 1904 [50]. Topology optimization, at its simplest, is a choice of design variables that allows for a prediction of a general distribution of material in space. [48] The truss origins of topology optimization are analogous to the individual elements used to represent the choice of materials in an optimized structure when in the context of tessellations used in finite element solvers, while any property can be theoretically be optimized before, much of topology optimization research focus on mass-minimization techniques for a given structural or conduction load. [38,48] The enhanced complexity offered by additive manufacturing techniques (with limitations [51]) have greatly expanded the possibilities of using topology optimized parts [38]; however, to date, there have been few examples of topology optimized parts in use. [38] Like with most modeling techniques of which topology optimization optimizes properties for, cellular material mechanics can often vary wildly from that of the assumptions used for isotropic materials, including the sensitivity of using certain structures over others for thin features optimized for. When sensitivities are taken into account, the physical realities of rough surface finish, stress concentrations during the manufacturing of large transitions in size of part features, and undesired tessellation from the topology optimization process can make the manufacture of these parts difficult. [38] From the export of a topology optimized surface to a CAD program to prepare for manufacture, it can be difficult to modify the topology optimized surface, leaving many topology optimization software to warn users that results should be used as a *guide* rather than a ready-to-use part design. [38] Finally, another limitation in layer-based additive manufacturing is the need of support structures for overhangs, some of which can require significant amount of post-processing to remove [38,51]. Mirzendehtdel and Suresh [51] developed an optimization algorithm to feed into a topology optimization for FFF of a polymeric part to minimize the support structure

required. There is a need to enhance the understanding of the mechanics and conduction properties of hybrid materials for topology optimization to be more useful to part designers.

Scientific Gaps and Research Plan

Scientific Gaps

As noted above, there exist several scientific gaps that limit the usefulness of hybrid material topologies from being used in application. The proposed work aims to develop design rules for hybrid materials for heat transfer and mechanical properties. The development of these design rules will require characterization and modeling.

The primary questions to address:

- How can existing tools be used to characterize hybrid materials with interpenetrating phases?
- How does the coherency of the bond between the hybrid material's constituents affect the resultant mechanical and heat transfer properties of the material?
- How does the geometry and surface roughness of the reinforcement phases affect the coherency of the bond? Do lattice configurations with higher surface areas per unit volume increase or decrease the coherency of the bond?
- How does geometry affect the overall tradeoffs in properties of a two-material hybrid produced with this print-then-infiltrate method? What are the design guidelines that prove that?

Material System

The material combination for the hybrid material studied is 316L stainless steel as the reinforcement with A356 aluminum alloy as the matrix. A356 and 316L were selected as the two constituents because A356/316L composites can have low densities, high thermal conductivities, and high toughness values, making them ideal for applications in which thermal and mechanical properties need to be simultaneously optimized (e.g., lightweight heat exchangers as described in the Motivation). This combination was chosen as the motivation for the work originated with engine pistons where Al-Si hyper-eutectoid alloys are the primary piston materials – A356, while being low strength at high temperatures and not used as a piston material, is highly castable, with properties that are well researched, while the 316L was chosen as a widely available powder used in powder-based laser

additive manufacturing with a higher ultimate strength and significantly higher ductility than the A356 alloy.

Experimental Methods

Part Fabrication

The hybrid A356/316L materials used for this work were prepared in two main steps. First, 316L preforms were fabricated using a Renishaw AM250 selective laser melting system with gas-atomized 316L powder feedstock (Fe-18Cr-12Ni-2Mo, wt%). The lattice geometry first used was a BCC structure, designed by Alex Pawlowski, as it was hypothesized by Keith Carver (ORNL) that this would provide the best opportunity for infiltration. A sprue and encasement were added to the geometry with input from Zachary Cordero (Rice). A lattice arranged in a BCC-like structure is made from 316L stainless steel powder using selective laser melting (SLM). SLM is a powder bed fusion additive manufacturing process where a fiber laser locally melts each successive powder layer with a programmed scan path. [38]

A spring-driven centrifugal casting machine was chosen as the infiltration method by Prof. Cordero as an inexpensive analog to pressure casting as gravity infiltration was found to be insufficient to fully infiltrate the steel preforms with molten aluminum. All metal infiltration was done at Rice University by Prof. Cordero. Alex Pawlowski was there to participate in the process of the metal infiltration, but due to the limited time there and safety protocol, Prof. Cordero performed the infiltration. The lattice boxes were mounted in plaster of Paris and pre-heated to 700°C in an induction oven. In the infiltration step, A356 is melted using an acetylene torch into a crucible containing the preheated 316L lattice box before the crucible is then spun in a Reymaster spincaster unit. As the crucible spins, the molten A356 flows into the 316L lattice box, fully infiltrating the box before the crucible stops rotating. This process provides an ease of manufacture; however, the cooling rate in the casting process is uncontrolled. In addition, the casting occurs in an open-air environment without a sprue for gases to escape; trapped residual gases as well as shrinkage pores were anticipated in the manufacture of the chosen material system.

The two-step processing strategy described above provides a solution to the difficulties of printing A356/316L composites with fusion-based additive manufacturing processes, which otherwise cause aluminum and iron to form brittle intermetallics [52].

Several preforms like the one shown schematically in Figure 3 were infiltrated. These preforms consisted of an open-cell mesh structure encapsulated by a 1 mm thick skin. The skin was useful because it served as both the ingate and mold during infiltration, thereby minimizing the need for additional tooling. The lattice region had body-centered cubic symmetry and dimensions of 40 x 40 x 13.5 mm³. Composites with different volume fractions of 316L were fabricated by infiltrating preforms that had the same unit cell size ($a = 2.5$ mm) but different strut diameters (D). The volume fraction of 316L and strut diameter of each composite are summarized in Table 1.

Mechanical Properties Determination

Existing characterization techniques used for hybrid materials are often techniques that have been extrapolated from monolithic materials that can be used to calculate bulk composite properties. In loading scenarios where the bulk property is not enough (in the case of when the wavelength of interaction is smaller than the periodicity of the hybrid material), the bulk assumption is not valid. Further insights into the spatial variation of properties is of vital importance for intended use.

Uniaxial compression and tension experiments were devised to determine the yield strength, ultimate tensile strength, toughness, and flow stress at various units of strain for the hybrid materials of various volume fractions. All compression and tension experiments were performed at ORNL by Shane Hawkins. Alex Pawlowski regrettably did not participate in the operation of the experiments but did create the drawings to get samples made at ORNL and in the data processing of the experimental results. Compression samples were in accordance with the ASTM e9 standard [10] for small specimens, a 10 mm diameter by 15 mm cylinder. As the lattice spacing used for the 316L reinforcement was 2.5 mm, this provided 4 base units of lattice material in diameter at 6 base units long. Tension samples were modified based on the ASTM e8 standard [9] as a compromise between the total length (38 mm) that was limited by the length of infiltrated material available (40 mm) and the cross section being large enough (4.5 mm) to be representative of the hybrid material with at least one repeating lattice base unit available. Due to the small size of the samples, and sensitivity to the lattice location, each tension sample was threaded into the grips. All tension and compression samples were cut perpendicular to the flow of the aluminum infiltration.

Heat Transfer Determination

In heat transfer, thermal diffusivity is the main measure of interest, as it directly compares the flow of heat from the hot side to the cold side. In the

motivation for the studied material systems, heat transfer is vital to the control of internal combustion processes. In a hybrid material of two very dissimilar thermal diffusivities, the characterization of the heat flow path is critical to understanding the applicability to given applications.

Thermal Diffusivity Experiment

Thermal conductivity measurements were conducted on blocks of infiltrated hybrid materials of each volume fraction of 316L by Ralph Dinwiddie (ORNL) using the single-sided transient plane source technique with a Hot Disk TPS 2500S instrument and a Styrofoam block as the thermal mirror [53,54]. Alex Pawlowski did not participate in the collection of the data. This technique records an average resistivity value of the measured sample that can then be inverted to a conductivity value. Because this resistivity is the sample average, it can only reveal the overall conductivity of the sample. For site-specific measurements of heat transfer, a thermal diffusivity calculation was made by the flash method [18] using the same setup used by Plotkowski, Dinwiddie, and Babu. [25] A 6000 J xenon flash lamp was positioned approximately 150 mm from one face of each sample. The pulse width was approximately 1 ms. A thermal imaging camera made by FLIR recorded the temperature rise at the surface of the opposing side to the side flashed for six seconds at a frame rate of 100 Hz. The approximate size of each pixel corresponded to 62.5 μm on the surface of the hybrid materials. An attempt to calculate the diffusivity at the site-specific regions was performed, with results in the below draft paper, assuming one dimensional heat transfer as per the assumptions made by the method. [18] The flash method described above directly measures the temperature on the side opposite of the face that receives the flash impulse from the xenon lamp. The rise in temperature is fit to an equation first devised by Parker et. al [20]. This procedure assumes an isotropic material in 1D heat conduction, with a characteristic path length of heat flow equal to the thickness of the sample. For site-specific measurements with a hybrid material, both assumptions are violated; in work in hybrid materials where a global average was calculated, it was assumed that the overall conduction could be considered 1D even though the conduction between the (often fiber-reinforced composites were studied) fiber and matrix was often 2- and 3-dimensional. Based on the apparent thermal diffusivity from each specimen (shown in the second paper in the Results chapter below), the 1D assumption is not valid, and most likely the apparent path length of heat transfer is much greater than the length of the sample. Modeling will be needed to add context to the heat transfer conditions to determine an appropriate apparent path length.

Simulation of the Flash Diffusivity Experiment

To add insights to experimental characterization, finite element modeling will be used. Using Abaqus, a closed-source finite element solver, one can reveal the interactions (simulated) inside of the material that we cannot directly observe with conventional techniques. While there are several other packages, some of which are open source, that could be used to solve the finite element problems, Abaqus is proposed due to the availability of resources to aid the modeling effort and the code that is expected. Additionally, a simulation – to then be validated – can provide more control over the variables that are involved, such as each constituent's composition, keeping the focus on the effect of the geometrical configuration on the material's spatial properties.

Chapter Three

RESULTS AND DISCUSSION

The following paper describes the manufacturing process and characterization of global mechanical properties and thermal conductivity of the produced hybrid materials and was published in Journal of Materials and Design in 2017. It is reproduced here with the permission of the publisher. Compression tests were performed to 50% of the original cylinder size for all volume fractions of hybrids produced, two tension tests were performed on 39 vol% 316L hybrid materials only, with one not reported in the paper below as it failed in the threaded section before considerable load was applied. Thermal conductivity was performed with a single side hot disk method described below that recorded an average thermal conductivity across the sample; no site-specific characterization was performed.

Statement of Work Performed by the Student

This paper and figures were written and placed together by Alex Pawlowski. There was, however, extensive help from Dr. Zachary Cordero of Rice University (listed as co-first author), as well as extensive review from the co-authors. Professor Cordero provided the SEM images and optical images in Figures 8 and 9. Matthew French (Rice University) mounted and etched samples and provided the optical image in Figure 2. Alex Pawlowski originally created Figure 1, 2, and 4. Ralph Dinwiddie (ORNL) performed the one-sided flash disk method to record thermal conductivity values of the various volume fraction samples. Keith Carver (ORNL) produced the SLM lattices using CAD files developed by Alex Pawlowski (with several iterations to get the format right for the SLM machine). Shane Hawkins (ORNL) performed the tension and compression tests used in this paper.

Damage-tolerant Metallic Composites Via Melt Infiltration of Additively Manufactured Preforms

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Highlights

A two-step processing route for fabricating metallic interpenetrating phase composites is described.

The composites exhibit tailorable thermal and mechanical properties as well as exceptional damage tolerance when loaded in tension.

Abstract

A356/316L interpenetrating phase composites were fabricated by infiltrating additively manufactured 316L lattices with molten A356. Measurements of the thermal conductivity of the composites showed an inverse rule-of-mixtures dependence on the 316L volume fraction. Compression tests revealed how the stress-strain response can be tailored by adjusting both the volume fraction and topology of the 316L reinforcement. Tension tests on composites with 39 vol% 316L showed a strain to failure of 32%, representing an order of magnitude improvement over the strain to failure of monolithic A356. Inspection of the as-

tested tensile specimens suggests that this exceptional damage tolerance is a result of the interpenetrating structure of these composites. These results together demonstrate that this two-step processing route avoids problems with intermetallic formation, cracking, and poor resolution that limit current fusion-based additive manufacturing techniques for printing metallic composites.

Introduction

Recently developed metal additive manufacturing techniques such as direct metal deposition [55,56], selective laser melting [57–61], and electron-beam additive manufacturing [62–65] use a high-power laser- or electron-beam to fuse feedstock, layer-by-layer, into a bulk product. These processes have attracted much interest from industry because they can print complex objects that are impossible to fabricate with traditional manufacturing techniques. Examples of additively manufactured components that highlight this exceptional control over form include lightweight robotic components [66] and advanced orthopedic implants [67,68].

Another unique capability of these direct metal additive manufacturing processes is that they can print net-shaped composites and functionally graded parts [69–72]. Historically this local control over composition has been achieved by changing the composition of the feedstock during printing [73,74]. However, this approach has two major drawbacks that have limited its usefulness. The first is that this approach has poor resolution and can only pattern the constituents on the scale of several millimeters. The second drawback is that the feedstock materials can react and form brittle intermetallics that subsequently crack due to the thermal stresses generated by the heat source [75,76]. Hofmann and coworkers have attempted to suppress intermetallic formation by tailoring the composition gradient to avoid intermetallic phases [74–76]. However, it is difficult to extend this alloy design approach to many materials systems.

Both problems stem from the fact that the mixing and reactivity of the constituents is promoted inside the melt pool. Here, we demonstrate a simple two-step approach for printing metallic composites that excludes such liquid-phase mixing and thereby overcomes issues with intermetallic formation, cracking, and poor resolution. In the first step of this approach, selective laser melting is used to fabricate a preform that serves as the skeleton of the component. In the second step, this preform is infiltrated with a liquid metal that has a melting temperature lower than that of the reinforcement. The resulting part is an interpenetrating phase composite (IPC) in which each constituent forms a continuous network. Melt infiltration of 3D printed preforms has previously been used to fabricate polymer/polymer [77] and metal/ceramic IPCs [40,78,79] but here we use this

approach to produce the first additively manufactured metal/metal IPCs. These metallic composites exhibit tailorable thermal and mechanical properties as well as exceptional damage-tolerance when loaded in tension.

Experimental Methods

A356/316L composites were prepared in two main steps. First, 316L preforms were fabricated using a Renishaw AM250 selective laser melting system with gas-atomized 316L powder feedstock (Fe-18Cr-12Ni-2Mo, wt%). Second, these preforms were mounted in plaster of Paris, pre-heated to 700 °C, and infiltrated with molten A356 (Al-7.6Si-0.25Mg-0.2Fe, wt%) using a spring-driven centrifugal casting machine. A356 and 316L were selected as the two constituents because A356/316L composites can have low densities, high thermal conductivities, and high toughness values, making them ideal for applications in which thermal and mechanical properties need to be simultaneously optimized (e.g., lightweight heat exchangers). Additionally, the two-step processing strategy described above provides a solution to the difficulties of printing A356/316L composites with fusion-based additive manufacturing processes, which otherwise cause aluminum and iron to form brittle intermetallics [52].

Several preforms like the one shown schematically in Figure 3 were infiltrated. These preforms consisted of an open-cell mesh structure encapsulated by a 1 mm thick skin. The skin was useful because it served as both the ingate and mold during infiltration, thereby minimizing the need for additional tooling.

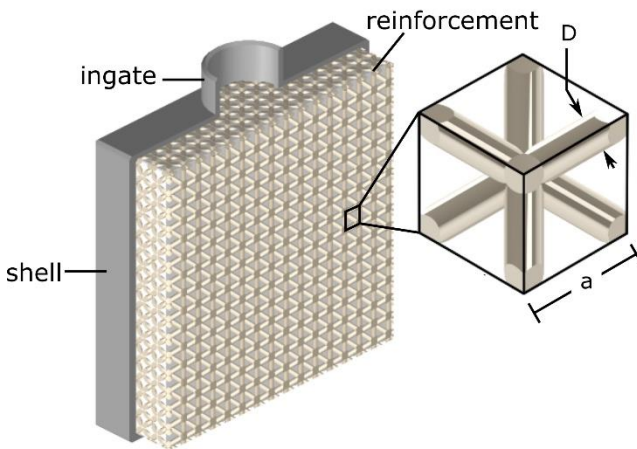


Figure 3 Model of additively manufactured preform. The unit cell of the reinforcement structure has an edge length a and contains four struts with diameter D .

The lattice region had body-centered cubic symmetry and dimensions of 40 x 40 x 13.5 mm³. Composites with different volume fractions of 316L were fabricated by infiltrating preforms that had the same unit cell size ($a = 2.5$ mm) but different strut diameters (D). The volume fraction of 316L and strut diameter of each composite are summarized in Table 1.

In order to characterize the microstructure of the infiltrated specimens, they were mounted in epoxy and polished using standard metallographic techniques. The porosity was measured using optical microscopy. The specific gravity of the infiltrated specimens was calculated by independently measuring their mass and volume.

After removing the 316L skin by face milling, the thermal conductivity of the composites was measured with the single-sided transient plane source technique using a Hot Disk TPS 2500S instrument and a Styrofoam block as the thermal mirror [53,54]. The reported values are the average of five measurements taken with a 0.8 W heat pulse and a 1 second measurement time.

Mechanical test specimens were harvested from the infiltrated composites using electro-discharge machining. The mechanical properties of the composites were evaluated using uniaxial compression and tension tests performed on a servo-hydraulic MTS 312.21 that was operated under displacement control. The compression specimens were cylindrical with a 15 mm height and a 10 mm diameter, giving a length: diameter ratio of 1.5. The compression tests used an initial strain rate of $\sim 5 \times 10^{-5} \text{ s}^{-1}$. The compressive strain was measured with an extensometer affixed to the compression platens. For comparison purposes, compression tests were also performed on fully-dense 316L cylinders prepared by selective laser melting as well as A356 samples harvested from an as-cast ingot. The tension specimens were round and had an 18 mm gauge length and a 4.5 mm gauge diameter. The tension tests used an initial strain rate of $3 \times 10^{-4} \text{ s}^{-1}$, and the tensile strain was measured using a clip-on extensometer. Tension tests were also performed on as-cast A356 specimens.

Table 1 Mechanical and material properties of the infiltrated composites.

Volume fraction 316L	Strut diameter, D (mm)	Specific gravity	Porosity (%)	σ_y (MPa)	n
0.29	0.72	4.10 ± 0.05	5.7 ± 0.2	140	0.26
0.39	0.83	4.61 ± 0.05	6.9 ± 0.2	150	0.31
0.46	0.91	4.95 ± 0.05	6.7 ± 0.2	155	0.33
0.54	0.99	5.67 ± 0.05	4.1 ± 0.2	250	0.32

The as-tested tensile specimens were inspected in a FEI Quanta 400 scanning electron microscope operated at 10 kV.

Results and Discussion

The porosity and specific gravity of each composite are summarized in Table 1. A representative optical micrograph of a cross-sectioned and polished A356/316L composite containing 39 vol% 316L is shown in Figure 4. The 316L and A356 materials exhibit distinct pore morphologies and microstructures that reflect their different processing histories. Specifically, the 316L material contains small spherical pores that are often found in additively manufactured materials [80], whereas the A356 material contains larger irregularly-shaped pores frequently observed in castings of Al-Si alloys [81,82]. Importantly, the 316L and A356 are separated by a gap with a mean thickness of $\sim 50\ \mu\text{m}$. Furthermore, SEM investigations on the A356/316L interface showed no evidence of Fe-Al intermetallic formation. The poor bonding between the 316L and A356 means the 316L reinforcement is unconstrained, which may improve the toughness of these composites, as described by Ashby et al. [83].

The thermal conductivity measurements are summarized in Figure 5, and they show the thermal conductivity decreasing with increasing volume fraction of 316L.

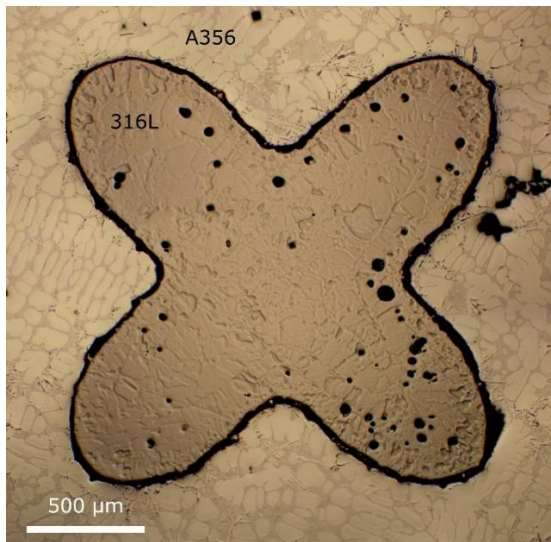


Figure 4 Optical image of an infiltrated composite revealing the isotropic dendritic structure in the A356 as well as the distribution of porosity in each of the constituents and at their interface.

Also shown in Figure 5 is the thermal conductivity predicted by an inverse rule of mixtures model:

$$K = (1 - P) \frac{K_{316L} K_{A356}}{f K_{A356} + (1 - f) K_{316L}} \quad (6)$$

where f is the 316L volume fraction, P is the porosity (which we set equal to 6% based on the results in Table 1), and K , K_{316L} , and K_{A356} are the thermal conductivities of the composite, 316L, and A356, respectively ($K_{A356} = 150 \text{ W m}^{-1} \text{ K}^{-1}$ [84]; $K_{316L} = 16 \text{ W m}^{-1} \text{ K}^{-1}$ [85]). The factor $(1-P)$ in Equation 6 accounts for the fact that at low temperatures, the porosity does not contribute to the thermal conductivity of the composite. There is good agreement between the experimental measurements and the thermal conductivities predicted by Equation 6, suggesting Equation 6 can be used as a guide to design A356/316L composites with specific thermal conductivity values.

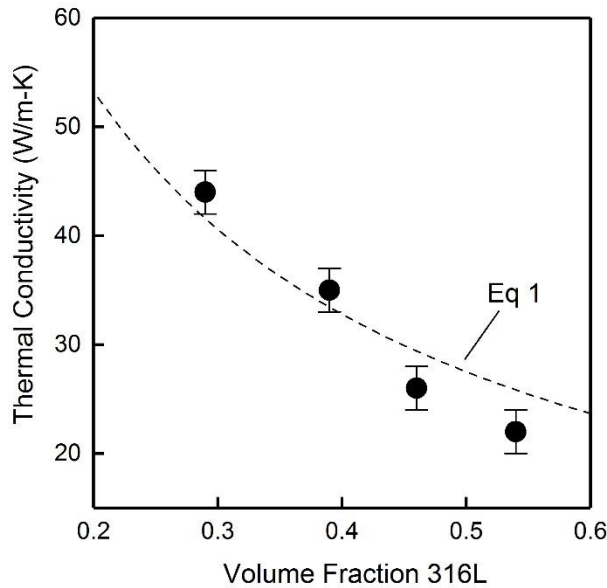


Figure 5 Thermal conductivity of the infiltrated composites. Analytical predictions are plotted as a dashed line using the properties of 316L ($K_{316L} = 16 \text{ W m}^{-1} \text{ K}^{-1}$) and A356 ($K_{A356} = 150 \text{ W m}^{-1} \text{ K}^{-1}$).

To quantify the mechanical properties of these composites, we performed uniaxial compression and tension tests on the as-infiltrated materials. Representative compressive true stress-strain curves for each of the infiltrated materials are shown in Figure 6a. The 0.2% offset yield strength, σ_y , and strain hardening exponent, n , of the composites are summarized in Table 1 and are seen to increase with 316L volume fraction.

In addition to changing the volume fraction of the constituents, another approach to controlling the mechanical properties of these composites is to tailor the topology of the reinforcement. Although we only characterized composites with body-centered cubic symmetry, our results provide some insight into how the distribution of the reinforcement can affect the compressive stress-strain response. Figure 6b shows experimental compressive stress-strain curves for monolithic A356, monolithic 316L, and the 39 vol% 316L composite along with a stress-strain curve for a 39 vol% 316L composite predicted using an iso-strain model and the stress-strain data of the constituents. The predicted stress-strain curve lies above the corresponding experimental stress-strain curve, suggesting that in the actual composite, there is appreciable strain localization in the weaker A356.

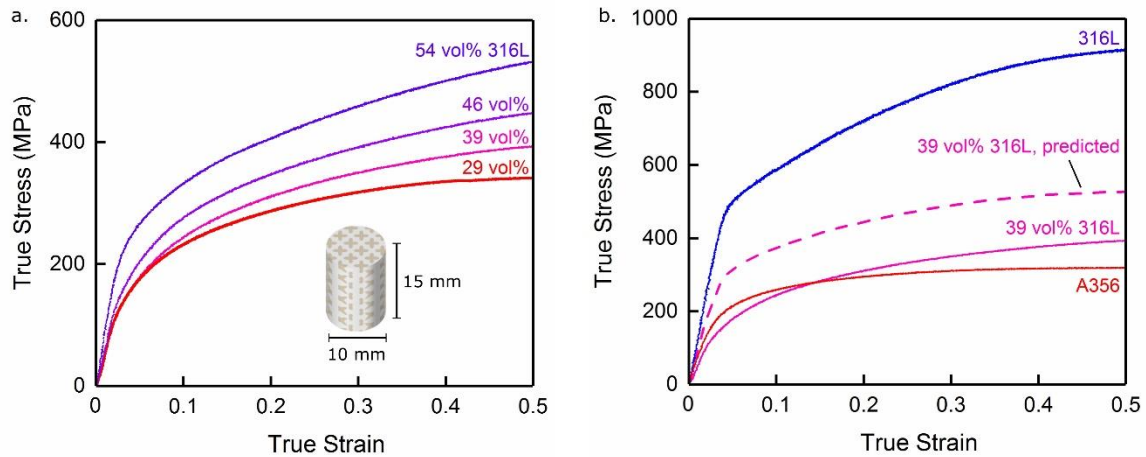


Figure 6 a) True stress-strain curves from compression tests on composites containing various volume fractions of 316L. The composites were oriented such that the edges of the unit cell were parallel with the loading axis. b) True stress-strain data from compression tests on monolithic 316L, monolithic A356, and the 39 vol% 316L composite. Also shown is a stress-strain curve for a 39 vol% 316L composite predicted using an iso-strain model and the stress-strain data of the constituents.

This interpretation is supported by Figure 7, which shows a cross-section micrograph of a composite specimen compressed to 25% plastic strain. Because the morphology of the dendrites in the A356 corresponds to the cumulative plastic strain, with more severely distorted dendrites indicating higher amounts of plastic strain, Figure 7 clearly reveals non-uniform plastic flow in the A356 as well as regions of especially high strain where A356 appears to have been extruded from between the 316L struts. This is in contrast with the relatively undistorted structure and pore morphology in the 316L material.

The strain localization and, relatedly, small contribution of the 316L material to the composite's flow stress, may be consequences of the fact that the reinforcement is patterned in a bending-dominated lattice, which can accommodate large macroscopic plastic strains under low applied stresses [86,87]. Thus, one potential strategy for increasing the strength of these composites (without changing the 316L volume fraction) is to pattern the reinforcement in a stretch-dominated lattice, an example of which is the octet-truss configuration. In this configuration, the 316L would be geometrically constrained to carry a higher fraction of the compressive load. More generally, this discussion serves to highlight how this processing strategy can be used to control both the volume fraction and the topology of the constituents to achieve specific mechanical properties.

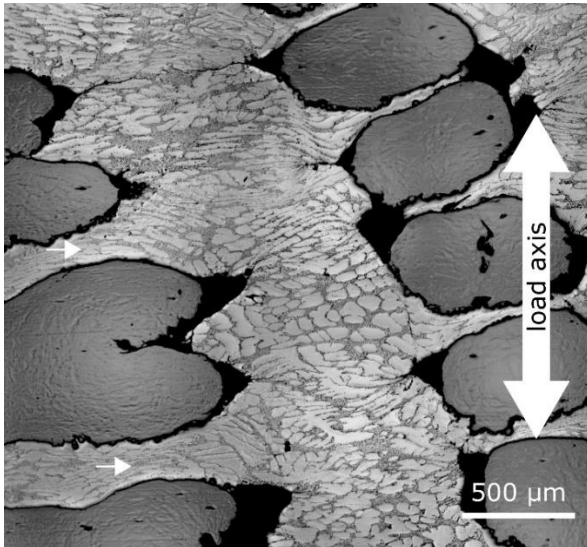


Figure 7 Optical image of a 39 vol% 316L specimen compressed to 25% strain showing the non-uniform plastic deformation and strain localization in the weaker A356. The white arrows indicate regions of especially high plastic strain in the A356.

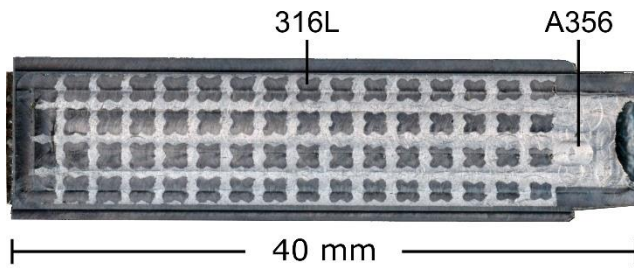


Figure 8 Optical image of a cross-sectioned infiltrated composite showing the 316L in dark contrast and the A356 in bright contrast. The volume fraction of 316L decreases from 0.7 to 0.2 going from left to right.

Another unique capability of our two-step processing strategy is that it can precisely tailor the structure over multiple length scales. The preform shown in Figure 3 contains a lattice with a constant unit cell size and strut diameter, but these parameters can change with position in order to print functionally-graded preforms. For instance, Figure 8 shows the cross-section of an infiltrated composite in which the strut diameter, and, as a result, volume fraction of 316L, decrease continuously from left to right.

The results shown in Figure 5 and Figure 6, together with the ability to print compositional gradients, hint at the possibility of using this two-step processing route to fabricate topologically optimized components in which the constituents are patterned for optimal performance. For instance, with this specific couple, regions of a component requiring high strength could be reinforced with 316L, while other regions requiring a high thermal conductivity could incorporate a high-volume fraction of A356.

We evaluated the damage-tolerance of these composites by performing tension tests on round specimens with a 4.5 mm gauge diameter. Figure 9 shows a representative tension stress-strain curve for a composite containing 39 vol% 316L. As a benchmark, a tension stress-strain for monolithic A356 is shown as well. The strain to failure of the monolithic A356 specimen is only 3%, in line with values reported in the open literature [88,89]. In contrast, the total strain to failure of the composite is 32% at an ultimate tensile strength of 95 MPa. The much higher strain to failure of the composite material is evident in Figure 10a, which shows the as-tested A356 and composite tensile specimens side-by-side.

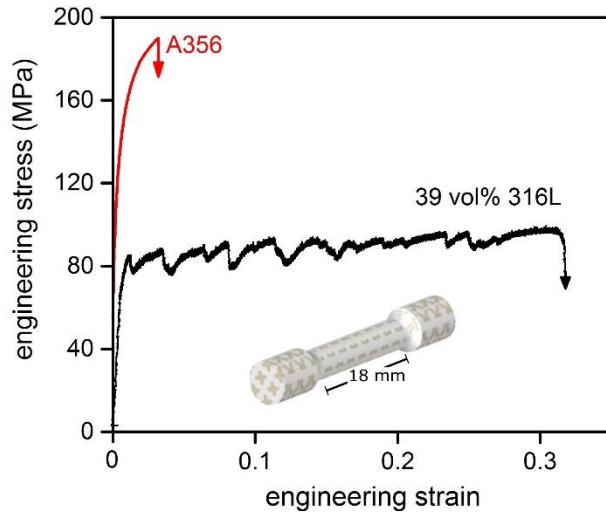


Figure 9 Engineering stress-strain curve from tension tests on a 39 vol% 316L composite and monolithic A356. The dips in the curve of the composite material correspond to cracking events in the A356. The composite tensile specimen was oriented such that the loading axis was parallel with the edges of the unit cell.

The surface of the composite tensile specimen in Figure 10a shows a number of cracks perpendicular to the loading axis. The serrations in the stress-strain curve of the composite specimen correspond to the formation of these cracks, which initiate when the A356 detaches from the 316L. Visual inspection of these cracks, of which an example is shown in Figure 8b, reveals that they are bridged by 316L struts. It therefore seems that the composites can accumulate far more damage and achieve a much higher strain to failure than monolithic A356 because of microcracking and crack-bridging, phenomena often observed in fiber-toughened ceramics but less frequently encountered in metallic systems.

Close inspection of the fracture surface of the composite tensile specimen shown in Figure 11a reveals how the cracks follow a tortuous path, propagating along the 316L/A356 interface as well as through the A356. Figure 11b, for instance, shows a decohesion zone adjacent to a region where the A356 failed by cleavage. This tortuous crack path results from the interpenetrating morphology of the two constituents [90]; if the constituents were not interpenetrating, the crack could have remained localized in the A356, causing the sample to fail in a macroscopically brittle manner at low plastic strain. Thus, the exceptional damage tolerance of these composites is likely a consequence of their interpenetrating structure.

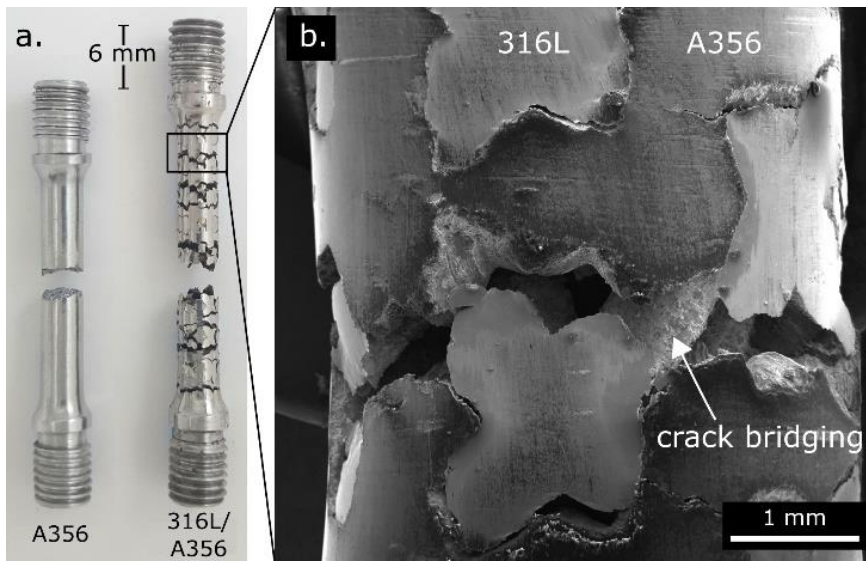


Figure 10 a) Optical image comparing as-tested A356 and composite tensile specimens that had the same initial dimensions. Note the much larger strain to failure of the composite and the extensive microcracking on its surface. b) Secondary electron micrograph showing a crack in the composite specimen that is bridged by the 316L reinforcement.

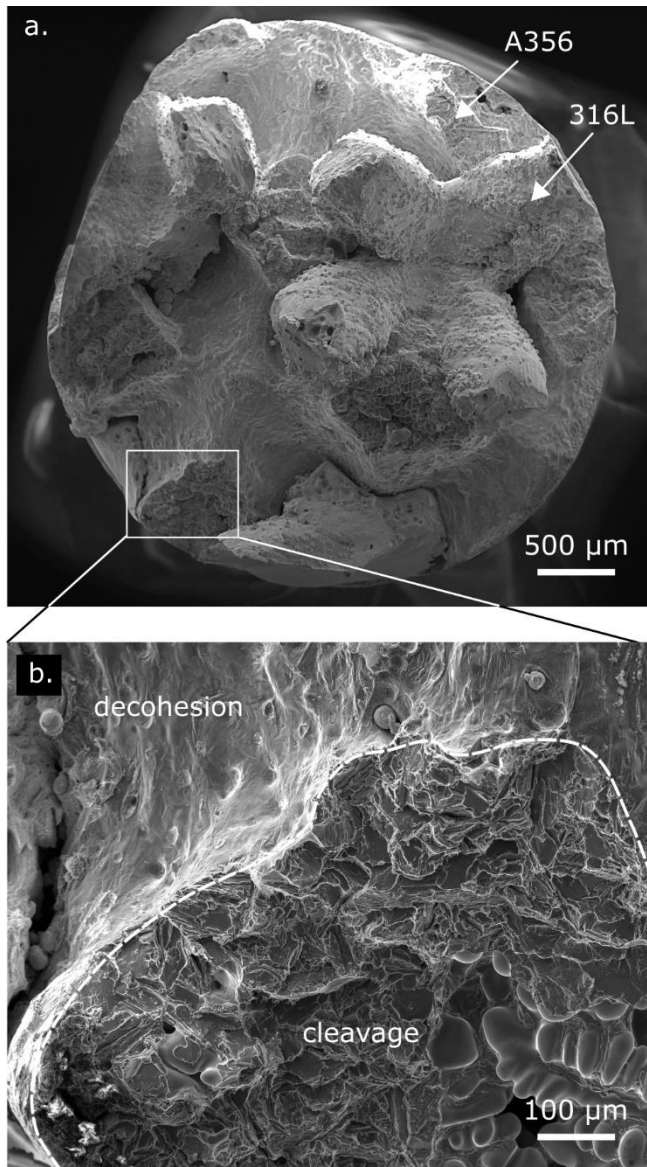


Figure 11 a) Electron micrograph of the fracture surface of the composite tensile specimen. b) Higher magnification image of the border between regions where the crack propagated through the A356 and along the interface between the component materials.

Conclusions

We have demonstrated a new approach for 3D printing metallic interpenetrating phase composites with exceptional damage tolerance as well as tailorable thermal and mechanical properties. The key advantage of this processing strategy over other fusion-based metal additive manufacturing techniques is that in this two-step process, liquid-phase mixing of the constituents is excluded. As a result, we can overcome problems with cracking and poor resolution that limit most of the other fusion-based additive manufacturing techniques for printing composites.

The A356/316L composites made with this two-step approach exhibit several interesting properties including, perhaps most significantly, a strain to failure that is an order of magnitude greater than that of monolithic A356. Further, we have illustrated how to achieve specific properties by controlling the volume fraction and the topology of the reinforcement. This capability can be used to print functionally graded components as well as composites with thermal and mechanical properties tailored to specific applications. Finally, it is important to note that although this work focused on 316L/A356 composites, the processing strategy that we describe is general and can be extended to any other metallic system in which the constituents have dissimilar melting temperatures.

Acknowledgements

This project was supported by the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, Vehicle Technologies Office, as part of the Propulsion Materials Program. The authors would also like to thank Prof. Suresh Babu and Dr. Peter Tortorelli for critically reviewing this manuscript.

The preceding section detailed the manufacturing process as well as the initial characterization of the compressive and tensile properties of the 316L – A356 hybrids. Thermal conductivity – measured as a single value from a thermocouple in contact with the A356 of the hybrids through a thin Kapton layer – was found to more closely follow the lower bound of thermal conductivity expressed by Hashin-Strihman bounds [91] as an inverse rule of mixtures. Thermal conductivity was measured several times for each sample with the mean values and their standard deviation reported previously.

In the next section, a different experimental method [18] was used to record the transient heat conduction through the hybrid materials that also looked at the site-specific variation of thermal diffusion. For a single diffusivity measurement averaged across composite samples, the violations of the 1D case are often found in literature, [92–95], but are generally considered acceptable, as the hybrids

tested, are either layered or particle-reinforced. In this experiment, a finite element model was paired to the experimental results to rationalize the results where the 1D assumptions were violated. Computed tomography was also used in conjunction to cross-sectioning to consider the effects of porosity and contact resistance on the thermal diffusion through the hybrids.

Using a linear relation to the thermal diffusivity of a material to its porosity content, a representative porosity could be estimated in the A356 only regions of the heat flow in the material. This is compared to an interpretation of computed tomography (CT) that was taken of each volume fraction of larger specimen. The specimens used in the flash method are the remains of the specimens as CT imaged that had been cut to make samples for optical microscopy, and therefore provide a relatively valid comparison of porosity calculation. This paper concludes that modeling is needed to understand the interactions of heat transfer across the specimens for the flash method to return a useful estimate of the diffusivity across each sample; the assumed apparent path length of heat transfer for which the diffusivity is calculated from is not simply the length of the sample as assumed for a homogenous material with 1D heat transfer. [18] In the next section, apparent path length will be used as a proxy for where the 1D assumptions break down within the hybrid materials.

Statement of Work Produced by the Student

This paper and all figures were written and placed together by Alex Pawlowski. Ralph Dinwiddie (ORNL) assisted with the flash diffusivity measurement, including applying a carbon coating to the samples. The samples were left over from previous samples cut by Randy Parten (ORNL). CT imaging was performed by Laser Design, Inc. of Minneapolis, Mn, with all interpretations of the image stacks written in Python by Alex Pawlowski using the sci-kit image library for the purpose of CT reconstruction. Code to capture the recorded FLIR IR data and calculate the apparent thermal diffusivity was provided by Alex Plotkowski (ORNL), and then re-written in Python by Alex Pawlowski.

Investigating the Breakdown in 1D Assumptions in Flash Thermal Diffusivity of A356 / 316L Hybrid Materials

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Introduction

Hybrid materials offer an opportunity to explore material property combinations not otherwise achieved with monolithic materials [26]. To quantify their properties at a local level – not just in bulk – there is a need to develop deeper insights into existing measurement techniques, which often assume homogeneous properties for single materials. Hierarchical structures [30,31] offer a unique combination of mechanical properties at low densities, matched by few materials, but little has been studied with regards to the thermal properties of these hybrid materials. Much work has been done regarding the mechanical performance of different open-cell [12,14,86,87,96–99] topologies and closed cell [100,101] structures where the hybrid material is a combination of open air and the particular material of interest. For hierarchical structures where the hybrid combination is of two or more solids as performed using electron beam melting and spark plasma sintering [44], laser sintering and centrifugal casting [102–104], even less work has been done of their mechanical and thermal properties, and specifically the local properties of the constituents within the hybrid materials [105]. Previous works in interpenetrating phase composites – hybrid materials with two or more overlapping constituent phases – [34,35,90,106–109] have similarly focused on the bulk

mechanical properties, as well as in different preparation methods for metal-matrix composites, many of which are also interpenetrating [110,111].

Flash thermal diffusivity is a relatively simple experimental technique that can provide insight into the heat flow within a given material [20,112,113]. Flash thermal diffusivity is also an important non-destructive technique that can be used to image and find discontinuities in otherwise homogeneous materials that are represented as a local change in diffusivity [25].

In composites, flash thermal diffusivity has been used to find an average temperature of the samples, with the assumption that the technique originally formed by Cowan [112] for homogenous materials is valid for non-homogenous materials. For composites, the geometry of the different phases plays a large role in the applicability of the homogenous assumptions. In the experiments of Taylor [22] and Taylor and Proctor [92], it was found that in layered composites, a sample length 4-5 times that of the length of the repeating pattern were sufficient in measuring a bulk thermal diffusivity that was not directionally dependent so as to cause significant heat loss axially to the measured back surface.

The purpose of this work is to characterize the heat transfer through the hybrid manufactured samples using the flash diffusivity method. This method is then compared to a heat transfer simulation to determine the average path length as a result of interactions with the less conductive, reinforcing phase in increasing volume fractions of 316L.

Experimental Methods

Manufacture of 316L – A356 Hybrids

Hybrid structures were manufactured by using laser powder bed fusion (L-PBF) to additively manufacturing a SS316L open lattice, which was then infiltrated with A356 using a spincasting technique. SS316L lattices were manufactured with 29, 46, and 54 volume percent using the same methods first described by Pawlowski, Cordero et al [102]. The reinforcing phase design consisted of an open cell lattice with a BCC structure with a set unit cell size ($a = 2.5 \text{ mm}$) for all volume fractions. The lattice was printed on a Renishaw AM250 using SS316L gas atomized powder (Fe-18Cr-12Ni-2Mo, wt%) with a particle size distribution between 15 and 45 μm supplied from Renishaw. The lattice was then melt-infiltrated with A356 using spring-driven centrifugal casting in an open-air environment. This method ensures infiltration of the lattice from bottom to top. Following infiltration, samples were face milled to remove the outer 316L skin, leaving the 316L / A356 hybrid materials. The volume fractions of 316L for each sample were determined experimentally by recording the mass of each sample,

measuring the volume, and using an Archimedes method to determine the closed porosity within the samples. Additional porosity estimates were made with optical microscopy from samples within the same batch of produced materials. Cylindrical samples 10 mm in diameter and 15 mm long were cut using wire electro-discharge machining with the top and bottom of the cylinder parallel to the plane of the lattice. These samples were then imaged using X-ray tomography.

Micro-computed Tomography

Computed tomography (CT) is a nondestructive technique that can reveal the sample fraction of different features based on the absorptivity of a penetrating wave for each feature, most often revealed using X-rays, as was used for these samples. To estimate the extent of porosity throughout each composite, X-ray micro computed tomography (microCT) was performed using a RXSolutions DeskTom tomography and digital radioscopy system. The unit uses an X-ray tube – with a Tungsten source – with a variable accelerating voltage from 40 to 150 kV; in this analysis, 130 kV was used. As the samples are scanned, X-rays project a magnified image onto a scintillator that ejects photons on to a flat panel detector (1920 x 1536 pixels in this study) per slice. The RAW image stacks produced are from reconstructed radiographs from filtered back-projection. The voxel size of the resultant images for the specimens was between 15 and 20 μm . Each sample was rotated around its longitudinal axis at a rate of 12.5 frames per second.

The image stacks were processed with Python using the scikit-image library [114]. Image stacks were denoised using a bilateral filter. While the image stacks enabled 3D reconstruction, 2D methods were used to minimize computation time and provide direct comparison to optical microscopy results, as done in other CT evaluations [115]. Once the images were cropped around the region of interest in each individual slice, non-linear mean denoising enhanced contrast of the two-phase composite was performed. Markers were developed based on regional thresholding to separate the X-ray attenuation recorded in each slice between the background, the two phases and their pores and the interface. Due to uneven attenuation in the CT images, a rolling window that looked at neighboring pixels was applied that identified whether a pore marked as an interfacial pore was at the boundary of two phases or was in fact within a single phase. Pores within the 316L phase and A356 phase that were not originally segmented with thresholding alone were separated from their parent phase by analyzing their gradient with a Sobel transform [116]. The cleaned markers were then fed into a random walker segmentation [117] algorithm. These segmented and labeled images were then read by slice to calculate the volume fraction of A356, and 316L, as well as each of their pores and pores at the interface between both constituents.

Porosity was calculated as the sum of the areas of pores within each phase divided by the total area of the slice. Pores at the interface (a significant amount of the void space seen in the composites) were attributed separately from the two phases and included in the final calculation of porosity for each sample. The total porosity of each sample was recorded as the sum of the pores in each phase plus the pores of the interface divided by the total area (pores and solid regions) of the slice.

Flash Thermography

Flash thermography is a nondestructive technique that measures the thermal response of a given sample. The setup was similar to that used in Plotkowski, Dinwiddie, and Babu [25]. Each cylinder was cut into 3 – 3.5 mm discs (a thickness of about 1.5 times the repeating unit size of the 316L lattice). To enhance the emissivity of the samples, the surfaces normal to the heat source were coated with a submicron colloidal graphite spray. This coating enabled the samples to absorb more energy from the flash source and to radiate heat more effectively toward the IR camera without affecting their thermal transport properties. Samples were mounted, three at a time, in a low thermal conductivity sample holder that obscured stray light of the flash source from reaching the IR camera, which can add error to calculations near the sample edge. A 0.5 mm hole was drilled through the sample holder, about 10 mm away from the samples, to provide an optical signal indicating the time when the flash occurred. The flash source was a 6000 J xenon flash lamp positioned approximately 150 mm from one face of the sample. The flash duration was approximately 1 ms. A FLIR sc8501 – a mid-wave IR camera - was used to record the thermal response of the opposite face of the sample. The camera was equipped with a 50 mm lens and a short extender ring was used to reduce the minimum focus distance of the lens. The distance between the lens and the sample was approximately 28 cm. The recorded frame rate was 400 Hz, with imaging starting about 2 seconds before the flash, and ending 4 seconds after the flash.

IR Data Analysis

The video capture was decoded using FLIR's Atlas SDK with the script first produced in [25], and then exported for analysis using open source Python libraries. The video capture of the 29, 46, and 54 vol% 316L samples was split into three separate files for analysis, with additional background cropped to reduce extraneous IR information. Each pixel had 2400 frames of temperatures during the experiment. These streams of temperatures for each pixel were then normalized by finding the average temperature of the pixel location in the time before flash ($T_{\min}$) as 0, and the average temperature of the region in the time after flash ($T_{\max}$)

as 1. The rise in temperature for each pixel was then fitted to a model first developed by Parker [20] using a Nelder-Mead simplex algorithm [21] to minimize the error (equation 1 is reproduced here):

$$\frac{T(t) - T_{min}}{T_{max}} = 1 + 2 \sum_{n=1}^{\infty} (-1)^n (-n^2 \omega) \quad (1)$$

$$\text{where } \omega = \frac{\pi^2 \alpha t}{L^2}$$

In equation 1, L is the thickness of the sample, α is the thermal diffusivity to be solved for, t is the time at which the temperature, T , is evaluated, with 8 terms (n) of the summation expansion used to balance precision and computational time. The thermal diffusivity to be solved for is a function of the half rise time of the normalized temperature rise, $t_{1/2}$, a constant = 0.1388, k_0 , and the thickness of the sample, L as seen in equation 2:

$$\alpha = k_0 L^2 / t_{\frac{1}{2}} \quad (2)$$

Corrections can be applied to account for radiative and convective heat losses as first proposed by Clark and Taylor [118], and Cowan [112]; in the experiment, no temperature loss was observed in the samples during the 6 second video capture. As the simple thermal diffusivity model above used in ASTM standard E1461 [18] assumes a constant thickness of a homogenous sample, the resultant thermal diffusivity is an apparent diffusivity that is actually a combination of the A356, 316L, and porosity. This can be displayed as a map of the sample to highlight spatial disparities in apparent thermal diffusivity within each sample and in comparison, to other samples of different volume fractions of 316L. From a 2-dimensional perspective, the composites have three regions: A356 at the surface with 316L behind; 316L at the surface with A356 behind; and pure A356 regions that are a consequence of the openings in the BCC lattice arrangement. The pure A356 region was then separated with a combination of edge detection and thresholding for each of the samples. The pure A356 region could then be used to detect variations in thermal diffusivity due to porosity, mean path length, and serve as a guide to the applicability of a 1D assumption, in comparison to the known thermal diffusivity of bulk A356.

Finite Element Model

From the work of Carslaw and Jaeger [119] for conduction in composites, it was determined that it was not possible to produce an analytical model to represent the 3D conduction in the hybrid materials, so instead a finite element model was

produced in Abaqus to solve the Fourier law of heat conduction over a discretized representation of each volume fraction to compare to the results observed in the experiment, the properties used in the model are listed in Table 2. An adaptive meshing scheme was implemented in Abaqus using the same seed parameters between each volume fraction, with an average element width $1/15^{\text{th}}$ of the repeating unit of the lattice. This element size was chosen for the final analysis using an h-refinement technique suggested by Zienkiewicz, Taylor, and Zhu [122] by continuously remeshing with smaller seeds until the half rise time through the sample was consistent between successive remeshing, as there was no analytical solution to compare with to determine an appropriate error threshold as suggested by Çengel [123]. The models assumed no porosity in the A356 and 316L phases, with a contact resistance applied between them to simulate the poor contact across the interface boundary; porosity at the interface observed in the samples was omitted from the models. The contact conductance listed in Table 2 was a result of comparing the rise in temperature of the 316L and A356 phases in the model to the rise observed in the experiment. No appreciable cooling was observed from the samples during the 6 seconds of capture in the experiments, therefore, adiabatic boundary conditions were applied to all external faces for each model. The contact resistance – applied globally to all contact pairs – between the two phases and the input heat (as a percentage of a total of the lamp load) were modified so that the end face temperatures would match that observed in the experiment.

Table 2 Model Values Used in the FEM simulation

Property	Material	Value	Ref
Thermal Conductivity	A356	151 W/m ²	[120]
Thermal Conductivity	SS316L	16.2 W/m ²	[85,121]
Density	A356	2670 kg/m ³	[120]
Density	SS316L	8999 kg/m ³	[85,121]
Specific Heat	A356	963 J/K-kg	[120]
Specific Heat	SS316L	500 J/K-kg	[85,121]
Contact Conductance	-	0.30 W/K-m ²	[123]
Incident Heat Flux - lamp	-	34.37 x 10 ⁶ J/m ²	
Pulse time	-	1 ms	

Results

Tomography revealed that the largest contributor of porosity within the samples were voids at the incoherent interface between the A356 and the 316L constituents, as seen in Figure 12. On a per slice basis, the porosity within the 316L phases was an extremely small contributor to the total porosity of each sample. As the volume fraction of 316L of the samples increased, the measured porosity at the interface also increased, where the 54 vol% 316L sample had nearly 10% porosity at the interface between the 316L and A356, the porosity fraction in the A356 and 316L otherwise showed no general trend.

In the flash experiment, the temperature rise in each sample was recorded, and then segmented for the pixels that corresponded to 316L and A356 at the surface, as seen in Figure 13. A separate segmentation was not made for the interface due to the small width of visible interface found at the surface of each sample. As seen in Figure 13, the A356 regions rise in temperature first before the 316L, with the difference in apparent temperature rise decreasing as volume fraction of 316L increases. In addition to the reduced temperature rise with increasing 316L volume fraction, the difference in initial temperature rise diminishes between the A356 and the 316L phases, to where it is indistinguishable in the 54 volume % 316L (Figure 13c) hybrid material.

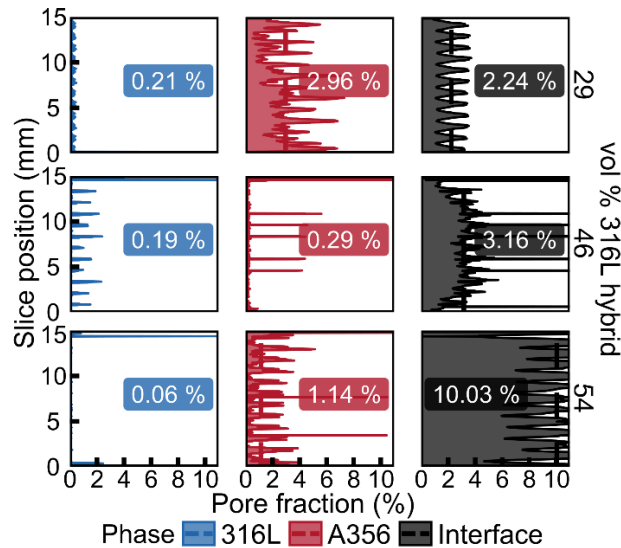


Figure 12 Computed Tomography of the 316L/A356 hybrid materials revealed that most of the porosity – defined as the area of pores within a phase divided by the total slice area - within each sample comes at the interface of the two constituents.

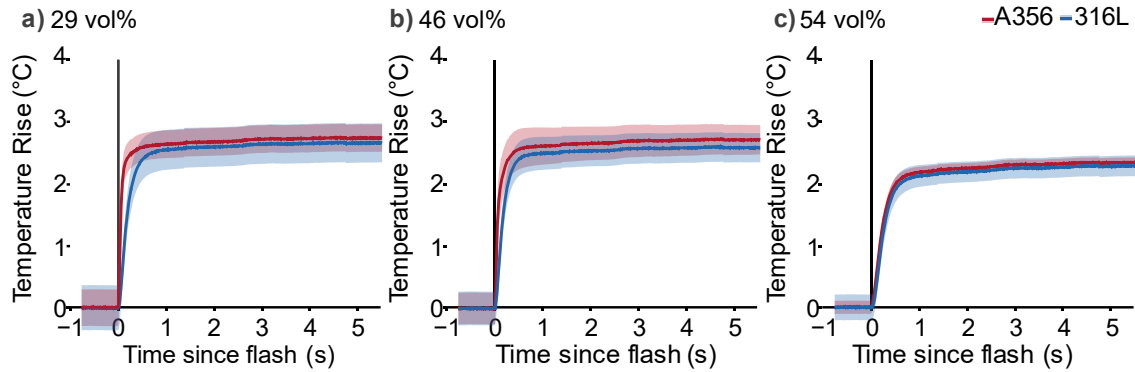


Figure 13 Mean temperature rise within each phase of each volume fraction sample

After the temperature rise in the sample is normalized to the mean minimum and maximum sample temperature, each pixel's temperature rise is then fit to equation 1 to determine the apparent thermal diffusivity for the pixel. Collectively, a map of apparent thermal diffusivity is then generated. The thermal diffusivity of cast A356 is $58.7 \text{ mm}^2/\text{s}$ [120], while the thermal diffusivity of the 316L is $4.06 \text{ mm}^2/\text{s}$ [85,121], as shown on the color bars of the apparent thermal diffusivity maps shown in Figure 14. The apparent thermal diffusivity in the regions where 316L is at the surface of the back face of each sample is higher than the thermal diffusivity of 316L, while the thermal diffusivity in regions where A356 at the surface of the back face of each sample is lower, and precipitously drops even lower as the volume fraction of 316L increases.

Due to the pattern of 316L (a BCC arrangement) used in the sample, as seen in the Figure 14, there are regions parallel to the flow of heat that are purely A356. These regions appear as brighter spots (most clearly seen in the 29 volume % 316L hybrid material in Figure 14a), as the thermal diffusivity is higher as the flow of heat has a one directional path unimpeded by 316L.

Discussion

As seen in Figure 14, the calculated apparent thermal diffusivity within regions of pure A356 are below that of the literature value of the thermal diffusivity of A356. Because the regions are unimpeded by 316L, factors that can affect the calculation of thermal diffusivity like porosity or length of heat transfer (originally assumed to be the same as the thickness of the sample) must explain the variation in thermal diffusivity observed.

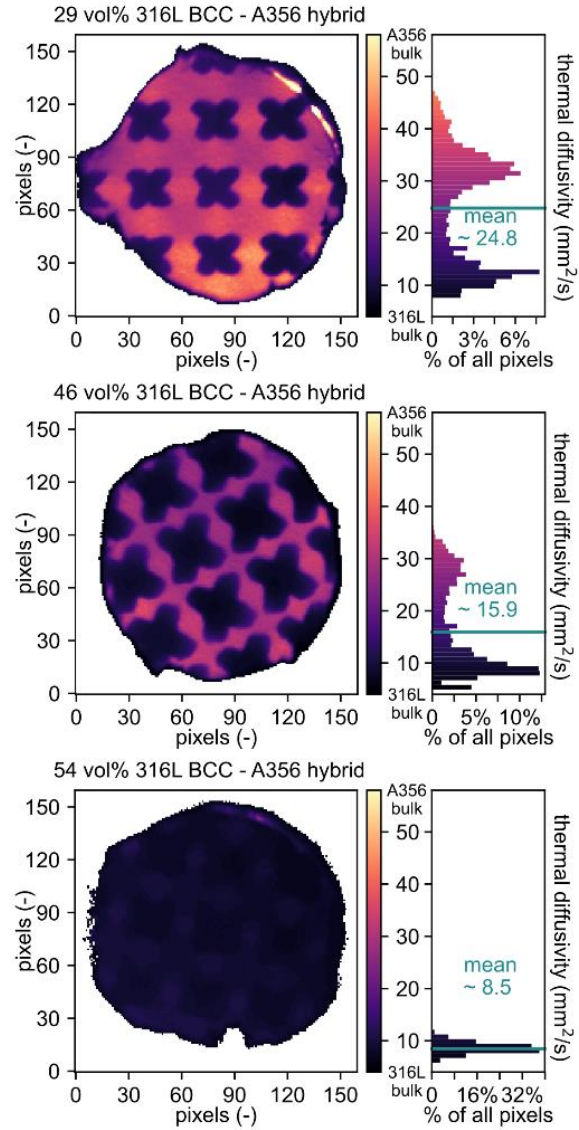


Figure 14 The calculated apparent thermal diffusivity pixel-by-pixel of each volume fraction of 316L/A356 hybrid material; as the fraction of 316L increased, the mean apparent thermal diffusivity decreased.

Loeb [124] first found a direct correlation to the change in thermal conductivity, and thermal diffusivity as a result of impedance by pores, over which radiative heat transfer and convective heat transfer would have to cross the pores. In simplification over many small pores, the change in thermal diffusivity is directly proportional to the amount of porosity found in the sample measured. Taking the calculated apparent thermal diffusivity and known thermal diffusivity of A356, porosity can be estimated using the following equation:

$$\alpha_{meas} = \alpha_{bulk}(1 - P) \quad (3)$$

The estimated porosity of each A356-only region for each volume fraction sample is shown in Figure 15. For even the lowest volume fraction (Figure 15a), the estimated porosity is significantly greater than the porosity found through tomography (Figure 12 vs Figure 15a). At the highest volume fraction, the estimated porosity is nearly over 80%, an estimate significantly higher than that recorded in tomography. It is extremely unlikely that the local porosity found within the smaller samples used in the flash thermal diffusivity experiments (cut from the samples imaged in tomography), greatly exceeds the average porosity found in tomography. Another explanation for this variation must be found.

In combining equations (2) and (3), the change in path length of heat transfer across the sample could help to explain the wide variation in apparent thermal diffusivity recorded, as seen in equation 4:

$$\alpha_{meas} = k_0 L^2 / t_{\frac{1}{2}} (1 - P) \quad (4)$$

In recalling that the original equation (1) assumes 1-D heat flow across the sample without porosity present, it is likely that the presence of pores within the A356 regions increases the apparent path length of heat transfer across the pure A356 regions. In Loeb's equation, the simplification of pores also presents a unique size dependence that may be violated; the pores in the A356 region of these samples may be as large as the region of pure A356, not significantly smaller than. By using the mean porosity of A356 recorded in tomography (Figure 12), the calculated half-rise time founded at each pixel in minimizing equation 1, and the calculated thermal diffusivity, the apparent path length of thermal diffusion can be found at each pixel within the pure A356 regions:

$$\sqrt{\frac{\alpha_{lit}}{k_0(1 - P)} t_{\frac{1}{2}}} = L \quad (5)$$

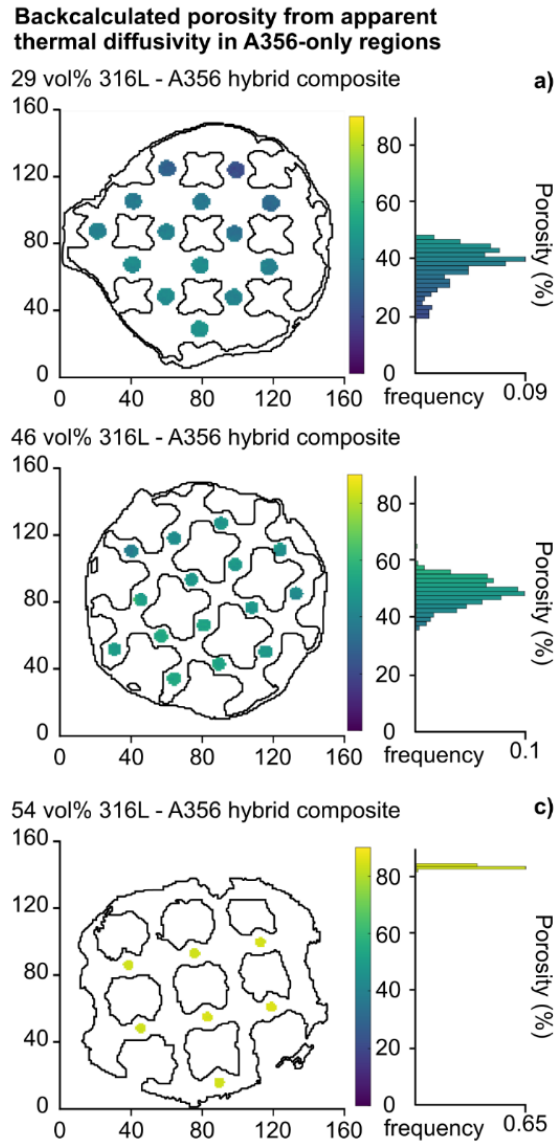


Figure 15 Porosity calculated from the difference in apparent thermal diffusivity from the thermal diffusivity of pure A356.

Figure 16 shows the estimated apparent path lengths in relation to the sample thickness. For the 29 vol % 316L and 46 vol % 316L hybrid samples, the estimated apparent path length – assuming 1 dimensional heat flow - is about 40% greater than the sample thickness. For the 54 vol % 316L sample, the estimated apparent path length is about 2.5 times the sample thickness, either the pore sizes are significantly smaller (thereby increasing the mean apparent path length) than in either the 29 vol% 316L or 46 vol% 316L samples, or the change in volume fraction has a significant effect on how heat is transferred in the A356 pure regions.

To look at the heat transfer inside of the hybrids, a finite element model was needed to assess the apparent path length difference caused by the interpenetrating steel structure within the A356, in comparison to a model without pores present. Figure 17 in comparison to Figure 14, corroborates some of the apparent path length change as a result of the presence of the 316L phase even in the A356 only regions as the calculated apparent diffusivity for even the A356 regions is below that of the A356 bulk diffusivity. Within the models, the apparent thermal diffusivity is highest at the A356 only regions nearest the edges, where less interaction with the 316L is occurring.

To investigate how the 1-dimensional assumption for heat transfer is affected by the presence of the 316L, the rise in temperature of the nodes through the middle of the produced 54 vol % 316L-A356 hybrid model was used, as seen in Figure 18. As the heat pulse moves through the sample, significant bowing of the temperature contours occur, indicating that the heat apparent path length (normal to the temperature gradient) diverges from the 1-dimensional assumptions of the theoretical diffusivity model shown in the frame at $t = 0.002$ (flash occurs at 0.001s). The peak of the bow, as expected, is the centerline between two regions of the less diffuse 316L phase; however, as shown in Figure 6, the speed at which this temperature contours diffuses through the sample is still slower than in an A356-only sample, as the average apparent path length is increased by diffusion away from the centerline.

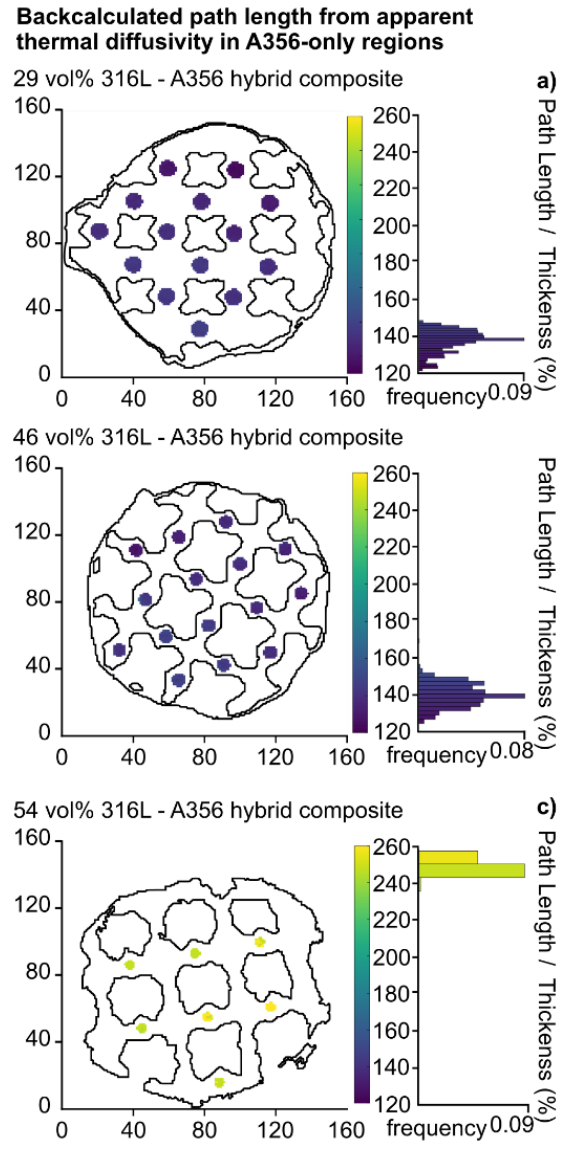


Figure 16 Estimated apparent path length of heat transfer divided by the sample thickness; the 29 vol% 316L (a) and 46 vol % 316L (b) hybrid materials show close agreement in about a 40% rise in apparent path length, while the 54 vol% 316L hybrid material (c); however, shows a drastic rise in apparent path length, of nearly 2.5 times the thickness.

To investigate how the thermal pulse is being affected by the presence of the 316L, the rise in temperature of the nodes through the middle of the produced 54 vol % 316L-A356 hybrid model was used, as seen in Figure 18. As the heat pulse moves through the sample, significant bowing of the temperature contours occur, indicating that the heat apparent path length (normal to the temperature gradient) diverges from the 1-dimensional assumptions of the theoretical diffusivity model shown in the frame at $t = 0.002$ (flash occurs at $0.001s$). The peak of the bow, as expected, is the centerline between two regions of the less diffuse 316L phase; however, as shown in Figure 6, the speed at which this temperature contours diffuses through the sample is still slower than in an A356-only sample, as the average apparent path length is increased by diffusion away from the centerline.

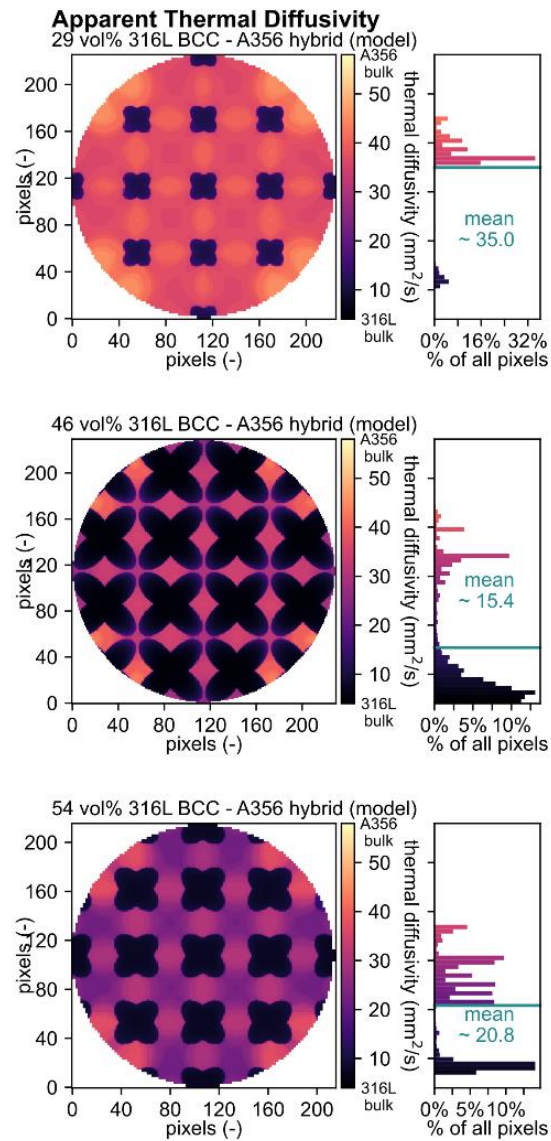


Figure 17 Calculated apparent thermal diffusivity values from finite element models of 29, 46, 54 vol % 316L - A356 hybrids

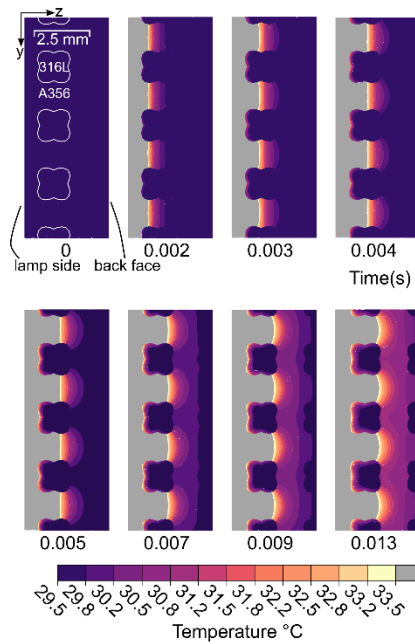
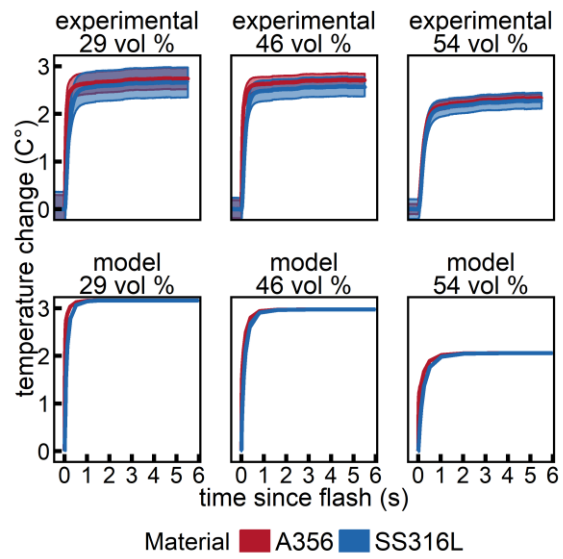


Figure 18 The change in geometry within the A356 (as a result of the 316L translates the uniform heat flux into point sources in the A356 only channels through a 54 vol % 316L - A356 hybrid.

The effect of porosity can be reexamined with a new apparent path length generated for the A356 only portions of each sample. The new apparent path length is calculated from comparing the diffusivity value for the A356 only regions with the diffusivity for the bulk A356 using equation 2. The recalculated diffusivity for the A356 only region is then compared to the anticipated porosity from equation 3. Figure 19 shows the percentage of porosity using the new apparent path lengths for each volume fraction. The median apparent porosity drops from nearly 40% to 13% for the 29 vol% and 46 vol% 316L – A356 hybrids, while the apparent porosity does not drop as dramatically for the 54 vol% sample. The result for the 54 vol% sample suggests that thermal diffusion through the sample is being further scattered significantly more than the simple assumptions for the reduced heat flow from pores would otherwise suggest. Of note, there were two A356 only regions within the 29 vol% sample that had a calculated diffusivity that exceeded the average diffusivity of the A356 only regions in the model, these regions were removed from Figure 19. While at cursory glance, the higher diffusivity value may suggest a thinner thickness in the local regions of the samples, or a cut that is off-axis to the repeating pattern of the interpenetrating structure, re-inspection of the sample shows no change in thickness or an off-axis portion of the lattice relative to the sides of the sample. Looking back at the model inputs, the contact resistance plays the largest role in the conduction between the 316L and A356. A larger contact resistance between the A356 and 316L decreases the half rise time in temperature of the A356, while increasing the half rise time of the 316L; increasing and decreasing the thermal diffusivities of the A356 and 316L respectively. Therefore, it is likely that the contact resistance is higher in this portion of the 29 vol% sample than elsewhere within the same sample, as a result of a greater void space between the 316L and the A356 as a result of the two-step manufacturing method [102]. By the same analogy, due to the higher surface area of the interpenetrating structure of the 54 vol % fraction relative to its volume, the contact resistance for the 54 vol % sample may be lower than the resistance found in the smaller volume fractions, increasing the apparent path length of the diffusion through the A356-only region. However, as shown in Figure 12, the measured porosity at the interface was significantly higher in the 54 vol% sample in comparison to the 46 vol % sample, which would otherwise increase the contact resistance. It is likely that the pores found at the interface for the 54 vol % sample are significantly large as to have an overall effect to slow the thermal diffusion through the sample.

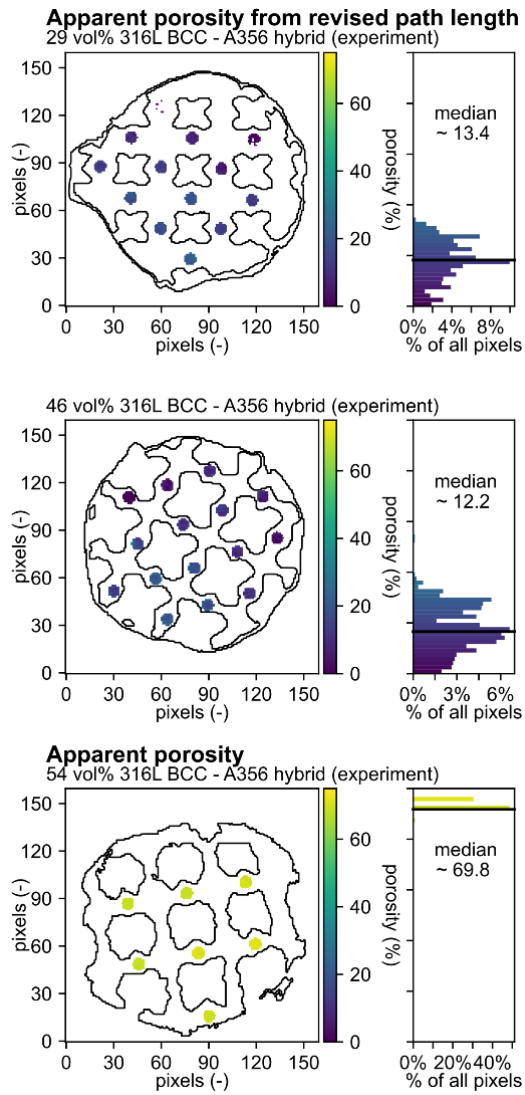


Figure 19 Apparent porosity in A356-only regions of 29 vol%, 46 vol%, and 54 vol% 316L-A356 hybrids as recorded in experiments, using the average apparent path length calculated from the apparent thermal diffusivity of the A356-only regions from the FEM models.

In addition, the relatively small change in surface area between 46 vol% and 54 vol% samples in comparison to the drastic difference in average diffusivities in the A356-only regions suggest that there must be a more complex interaction between the interface and porosity within the A356 to will need to be further investigated in a follow-on study.

To explain why the reduction in diffusivity is observed in the A356, we look at the CT of porosity in the sections of the diffusivity sample. The resolution of the CT image is 15 μm , but many of the pore diameters are below 15 μm as seen in the optical micrograph for each sample. A micro CT image is received as a map of intensities from the scintillator. This can be mapped to the linear attenuation coefficient for each pixel, which can be compared to standard attenuation values using the NIST XCOM database with parameters of the x-ray source used.

A conversion of the recorded intensity to a linear attenuation coefficient is done by taking a reference image from CT of an A356 only sample of low porosity, which contains both A356 and the background air, and scaling CT images from hybrid samples to the reference. The intensity of the A356 and the intensities of the air are used to fit a slope of intensity to attenuation. This map is then related to the different volume fraction samples by comparing the intensity of the background air in each sample to that of the reference and increasing or decreasing the entire image intensity to match. The image stacks for each sample can then be segmented to remove the background from the total count; and the centers of the regions where there is only A356 can also be segmented to reveal the average attenuation within those regions relative to the reference value. The attenuations can be compared between each sample as shown in Figure 20. The two peaks show the average attenuation within the A356 and the 316L. The count of each peak is directly related to the volume fraction of each sample, the location along the attenuation axis can be compared to the reference value of attenuation. At higher volume fractions, the attenuation within the A356 is closer to that of the reference value. In the optical sections to the right, the pores within the A356 of each sample are less numerous as the volume fraction of 316L increases; this directly correlates with the shift in the attenuation peak. Of note, the pores within the 316L also shift the attenuation peak to the left. In addition, pores larger than 2 or 3 pixels cubed (that would have a value close to that of air) are more numerous as well as pores that are smaller than 15 μm . Because there are pores smaller than the resolution limit, if both the A356 and 316L were segmented, there could be underlying pores that are missed within the CT.

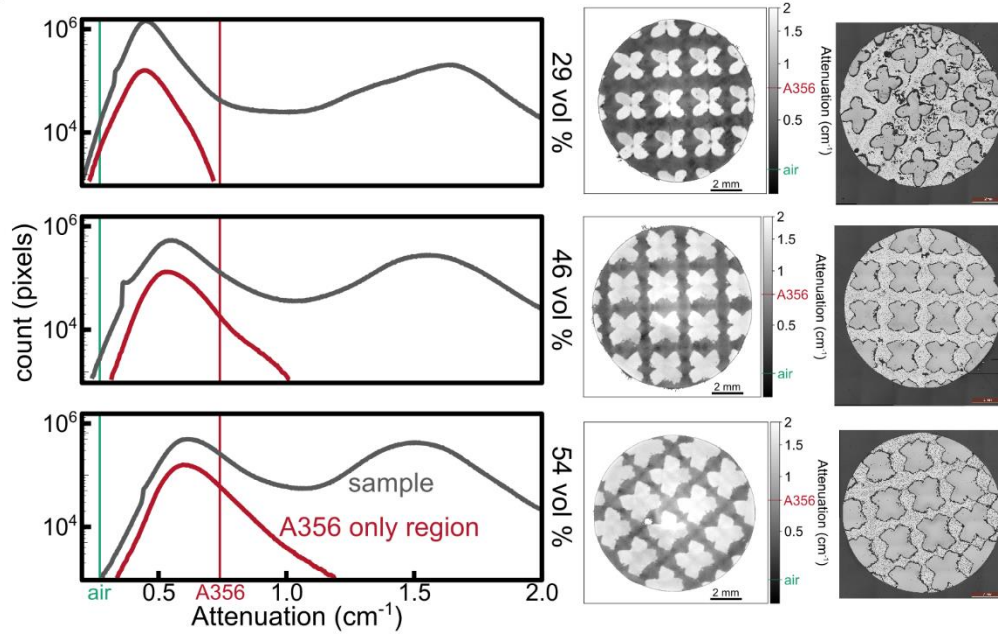


Figure 20 Attenuation in microCT images of each volume fraction sample compared to optical

Conclusion

The thermal diffusivity of hybrid 316L / A356 composites was investigated using flash thermal diffusivity to understand the local characteristics of the heat transfer through the composite. X-ray CT revealed that porosity at the interface of the two materials was the largest contributor to the total porosity within each volume fraction of hybrid material produced, with a smaller subset of porosity found within the A356 phase. Due to reinforcing nature of the 316L, the local thermal diffusivity across each sample varied widely from a maximum in the pure A356 regions to a minimum where the 316L was at the back surface of the material. In estimating the porosity from the reduction of thermal diffusivity, it was uncovered that the assumption of the sample thickness as the mean apparent path length of heat transfer did not match the measure of the pores found in each sample through X-ray CT. When using the porosity values calculated from X-ray CT, the mean apparent path length of the heat transfer through the samples were 40% longer than the sample thickness for the 29 vol% 316L and 46 vol% 316L samples, while the estimated apparent path length was 2.5 times the sample thickness for the 54 vol% 316L sample. In re-examining the locations of pores with respect to the A356 regions, the apparent path length increase as estimated is likely due to the variation in porosity across the sample for the 29 vol % and 46 vol % 316L hybrid materials. For the 54 vol % 316L material, the drastic reduction in thermal diffusivity

is most likely a consequence of diffusion that has become dominated by the much less diffuse reinforcing structure. To understand the heat flow at higher volume fractions and what interactions porosity within the phases and at the interface, a 3D simulation was needed. A finite element model simulated this variability in properties based on the position of the 316L phases. Due to the interaction between the A356 and the 316L interpenetrating structure, thermal diffusion is slowed even in heat path regions where no 316L is present. When comparing the thermal diffusivity in the A356-only regions within a finite element model for each volume fraction to the bulk diffusivity of A356, the resultant apparent path length change was between 1.2 to 1.4 times the sample thickness (increasing for increasing volume fractions of 316L). The apparent porosity for the 29 vol% and 46 vol% samples is then reduced to 13% using this larger apparent path length in comparison to nearly 40% when using the sample thickness as the apparent path length. The apparent porosity of the 54 vol% sample is still significantly larger than that calculated from the X-ray computed tomography or the lower volume fraction samples suggesting a complex interaction between the pores at the interface that overcomes the added surface area of the larger sample. When the intensity of the X-ray computed tomography was mapped to the attenuation of air and A356, it was revealed that the attenuation did not match the expected attenuation of A356, suggesting micropores smaller than the resolution size were present. This can help to explain the lower diffusivity as the presence of small pores further drops the direct diffusion path within the A356. Further investigation in a follow-on study will be needed to understand the role of the heat interaction with the presence of large pores at the interface between the 316L and A356; however, when the flash disk method is paired with a finite elements model, a revised mean apparent path length can be used to reveal the average defects within a composite.

Chapter Four

CONCLUSION

A new approach for 3D printing metallic interpenetrating phase composites was developed with appreciable damage tolerance as well as tailorable thermal and mechanical properties. The key advantage of this processing strategy over other fusion-based metal additive manufacturing techniques is that in this two-step process, liquid-phase mixing of the constituents is excluded. This helped to overcome problems with cracking and poor resolution that limit most of the other fusion-based additive manufacturing techniques for printing composites. The A356/316L composites made with this two-step approach exhibit several interesting properties including, perhaps most significantly, a strain to failure that is an order of magnitude greater than that of monolithic A356. Further, it was shown how to achieve specific properties by controlling the volume fraction and the topology of the reinforcement. This capability can be used to print functionally graded components as well as composites with thermal and mechanical properties tailored to specific applications. Although this work focused on 316L/A356 composites, the processing strategy can be extended to any other metallic system in which the constituents have dissimilar melting temperatures.

The thermal diffusivity of hybrid 316L / A356 composites was investigated using flash thermal diffusivity to understand the local characteristics of the heat transfer through the composite. X-ray CT revealed that porosity at the interface of the two materials was the largest contributor to the total porosity within each volume fraction of hybrid material produced, with a smaller subset of porosity found within the A356 phase. To understand the heat flow at higher volume fractions and what interactions porosity within the phases and at the interface, a 3D simulation was needed. A finite element model simulated this variability in properties based on the position of the 316L phases. Due to the interaction between the A356 and the 316L interpenetrating structure, thermal diffusion is slowed even in heat path regions where no 316L is present. When the intensity of the X-ray computed tomography was mapped to the attenuation of air and A356, it was revealed that the attenuation did not match the expected attenuation of A356, suggesting micropores smaller than the resolution size were present. This can help to explain the lower diffusivity as the presence of small pores further drops the direct diffusion path within the A356. Altogether, the flash disk method can be used to not only estimate the effective thermal diffusivity of the sample but also be used to characterize the amount of pore interaction within the sample, that may not be easily resolved with X-ray CT. When the experimental method is compared to a

finite element simulation, the deviations from the 1D thermal diffusivity can be normalized to the model, then used to indicate areas of heterogeneity caused by site-specific changes in porosity and/or defects in the hybrid material.

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