

Chapter 3

Interfacial Characterization of Metal Wire Inlays for 3D Printed FDM Parts



Vereesh Ayyagari, Hugh A. Bruck, Amir H. Ohadi, and Michael M. Ohadi

Abstract A novel process for creating 3D printed parts with metal wire inlay has been developed at the University of Maryland. By using metal wire inlay, parts can retain the properties of drawn metal wires that can be either embedded in polymer or fully exposed. As a result, the mechanical integrity of the metal wire interface with the FDM polymer can be controlled through the printing conditions and preparation of the wire surface. In this investigation, we develop a novel test specimen for characterizing the mechanical integrity of the metal wire/polymer interface. In particular, the test specimen is designed to quantify the ability of the interface to provide hermetic sealing when loaded by pressurized fluids. The effects of the processing and preparation conditions on the interfacial strength are also investigated via single fiber pullout tests. As a result, it is possible to design new 3D-printed metal-polymer composite structures with free standing metal wires that can be utilized in a variety of different applications ranging from integrated flow sensing to enhanced thermal control.

Keywords Metal-polymer composites · Polymer-metal interface · Failure characterization · Adhesion · Interfacial strength

Introduction

Polymer matrix-metal wire composite structures are increasingly being developed for strengthening polymer structures [1], heat exchangers [2], and conductive path tracing for electronic components [3]. For a mechanistic point of view, there could be two variants of the composites. In the first, the metal wire is wholly embedded in the polymer. In the second composite, the metal wires are partially exposed, resulting in a partially free-standing metal wire. The two variants of the composites are shown in **Figure 1**, where Figure 1a shows the polymer-metal composites where the metal wires are completely embedded into the polymer, and 1b shows the composites where metal wires are partially free-standing.

While the fabrication of the composite can be achieved through conventional manufacturing techniques, additive manufacturing, commonly referred to as 3D Printing, has become popular choice of fabrication, primarily due to the complex nature of the composite. For 3D printing of the composites, Fused Filament Fabrication (FFF) has been widely used for depositing the polymer, and metal wires are thermally embedded. In FFF, thermoplastic materials, such as polycarbonates, acrylonitrile butadiene styrene (ABS), Polylactic Acid (PLA), and Poly Amides (PA), are used as feedstocks due to their favorable properties such as low melting temperature and appropriate melt viscosities [4]. Recently, we have developed a 3D printing process for metal wire-inlay to create cross-media heat exchangers [5–8] which contains an array of free-standing metal wires embedded into the polymer structure.

The failure mechanisms of the polymer-metal composites are critical to understanding their limitations in the applications. In particular, to estimate how much pressure can be applied to the structure before the wires start pulling out i.e. interfacial strength between metal and polymer, wire push out tests are applied [1, 9–11]. While a few studies exist on the characterization of composites where metal fibers that are embedded continuously in a polymer matrix, very few have studied 3D-printed polymer-metal composites fundamentally, where the metal wires are partially exposed. The current study focuses on the failure characterization of the partially free-standing metal fibers in polymer-metal composites.

Vereesh Ayyagari · Hugh A. Bruck · Amir H. Ohadi · Michael M. Ohadi

Department of Mechanical Engineering, A. James Clark School of Engineering, University of Maryland, College Park, MD 20742
e-mail: veeresha@umd.edu; bruck@umd.edu; amir@umd.edu; ohadi@umd.edu

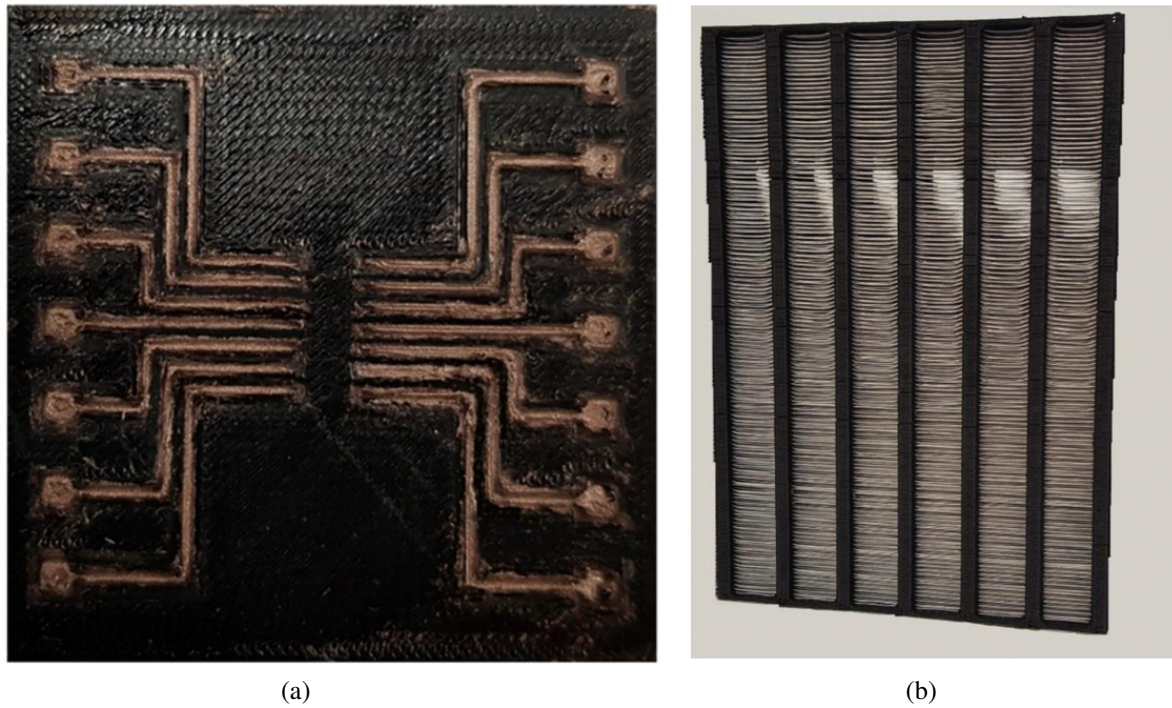


Fig. 1 The variants of polymer-metal composites: (a) continuously embedded metal fiber in the polymer [6], and (b) Partially embedded metal fibers in the polymer [2]

Specimen Fabrication and Testing

Polymer matrix-metal wire composites can be used as functional structures in a variety of applications requiring the conductivity of metal and the lightweight provided by polymer supports (e.g., lightweight heat exchangers). In many applications, the polymer structure may be pressurized, such as by a working fluid in a heat exchanger and may be subject to a differential pressure that may cause a partially free-standing metal wire to pull out of the polymer support structure. For this investigation, a specimen was designed and fabricated that allowed for testing of multiple partially free-standing wires embedded in a polymer structure that is subjected to a differential pressure (i.e., a polymer pressure vessel with metal stringers). The cross-section of the composite specimen that was fabricated and tested in this investigation can be seen in Figure 2.

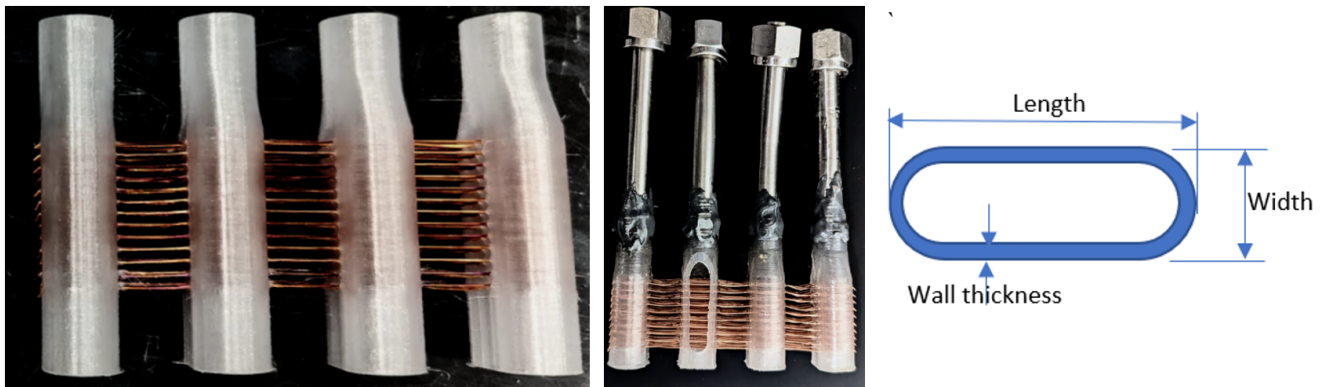
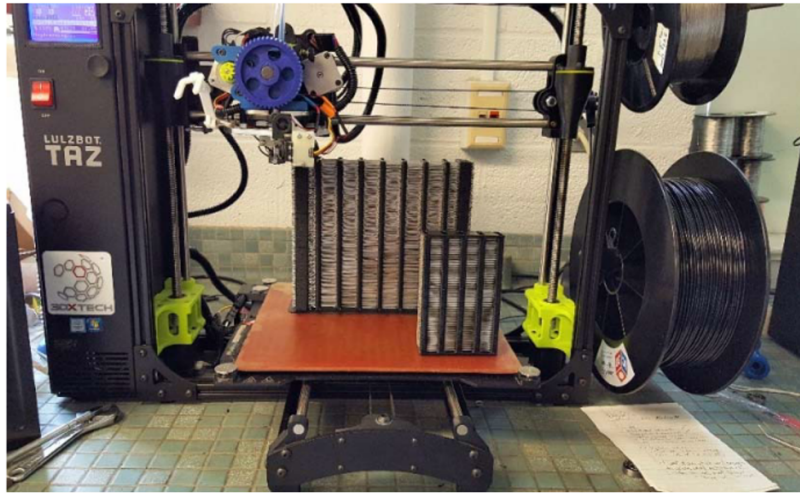


Fig. 2 (left) Cross-media coupons (middle) Cross-sectioned coupon exposing free-standing wires inside (right) The geometrical parameters of the cross-media coupons as seen from top cross-section view

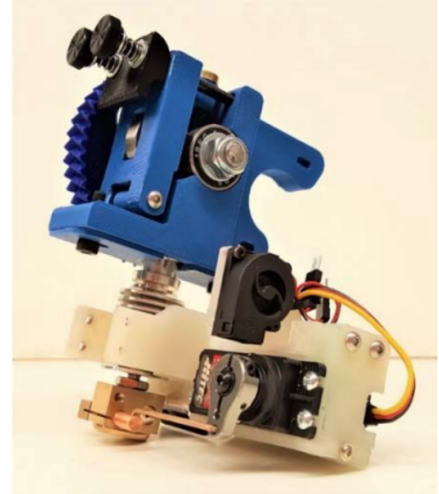
To maintain its integrity in applications where the polymer structure is pressurized, the composite structure should not leak at the metal wire/polymer interface during operation, i.e. maintain the sealing in pressurized condition. Currently, there

is no standard available for determining the maximum pressure limit for this type of specimen. Therefore, to obtain the pressure at which the leakage happens for different geometries, specimens were fabricated and tested using polycarbonate as the polymer, and copper wires as the metal fibers.

Specimen Fabrication: To fabricate specimens, an off-the-shelf polymer 3D printer was customized to embed metal fibers over the polymer layers with a proprietary metal fiber print head, shown in **Figure 3**. To facilitate the visualization of the metal wire/polymer matrix failure (i.e. leakage), clear polycarbonate was chosen as the polymer and copper as the metal. The coupons as printer were found to be leaking near the metal-polymer interface. To avoid this leakage, the composite was dip-coated in a commercially procured 3d print sealer, an epoxy-hardener mixture, and cured in an oven at 50 °C for 24 hours. A post-curing regimen was followed where the cured coupons were kept in an oven at 90 °C for 2 hours and allowed to cool in the oven slowly to minimize residual stresses.



UMD TAZ Printer



Proprietary Metal Fiber Print Head

Fig. 3 (left) A 3D FDM printer customized at UMD for metal wire inlay using (right) a proprietary metal fiber print head.

Two different types of cross-media coupons were tested for failure. All coupons had the same geometrical parameters except for wall thickness. The vertical and horizontal pitches were maintained the same in all coupons. The geometrical parameters of the cross-media coupons are mentioned in Table 1.

Table 1 Geometrical parameters of the cross-media coupons

Parameter	Value	
	Coupon-1	Coupon-2
Wall thickness (mm)	1.8	3
Length (mm)	20	
Width (mm)	11.5	
Wire diameter (mm)	0.5	
Vertical wire pitch (mm)	3	
Horizontal wire pitch (mm)	2.6	

All coupons after the post-curing were tested for leakage at 50 PSI using a bubble test. Only the coupons that passed the bubble test were used for failure characterization. For failure characterization, a pressure decay method was used. The schematic of the experimental setup is shown in **Figure 4**. To pressurize the coupon, an Additel 925 hydraulic pressure test pump was used. Once the unit under test (UUT) reaches the required pressure, the needle valve is closed to shutoff the UUT from the pump and allow for pressure decay. To record the pressure in the coupon a pressure gauge is connected between the valve and the UUT. A DAQ was connected to the pressure gauge to collect the pressure data every second to monitor the pressure decay.

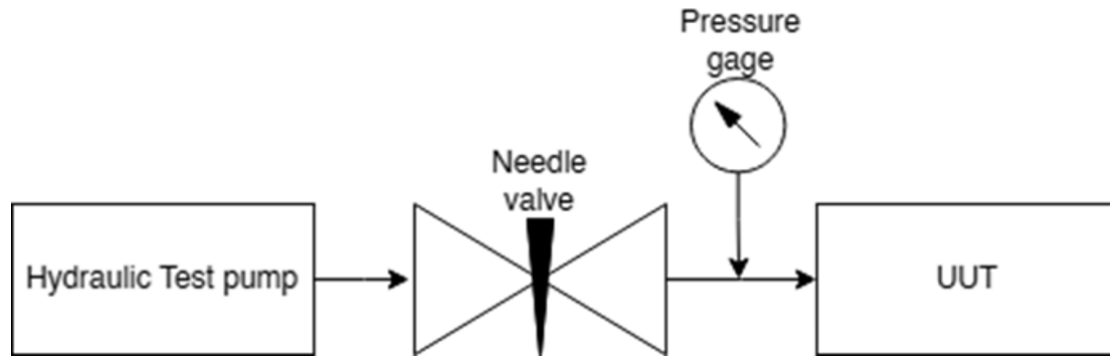


Fig. 4 Pressure decay test schematic

Experimental Results

Three specimens with the desired test geometry were fabricated and tested, as well as another two specimens that had slightly thicker polymer support. The resulting variation of pressure with time for one of the specimens with the thinner polymer supports is shown in **Figure 5**. The pressure decay test started at 50 PSI and was held for 10 minutes before being increased in 25 psi steps holding 10 minutes between steps, and was carried out at 25 PSI increments. At each pressure level, the UUT was shut off from the pump to obtain the pressure decay rate. At 275 psi, a water droplet was observed on the outer surface of the specimen at the metal wire/polymer matrix interface, indicating leakage, which was considered a catastrophic failure and ended the test. A similar testing protocol was carried out on two other specimens and three samples of coupon2 and the pressure decay rate at each pressure level is reported in **Figure 6**. As seen from the figure, until 150 PSI, the pressure decay rate stays relatively constant. However, the decay rate increases significantly with further increases in pressure above 150 PSI. This transition point at 150 PSI indicates that it should be considered the pressure limit. Additionally, the decay rates are different for each step above 150 PSI, which could potentially be attributed to the number of failure locations, since there are multiple metal wire/polymer support interfaces. For the second specimen geometry, the pressure decay rate transition

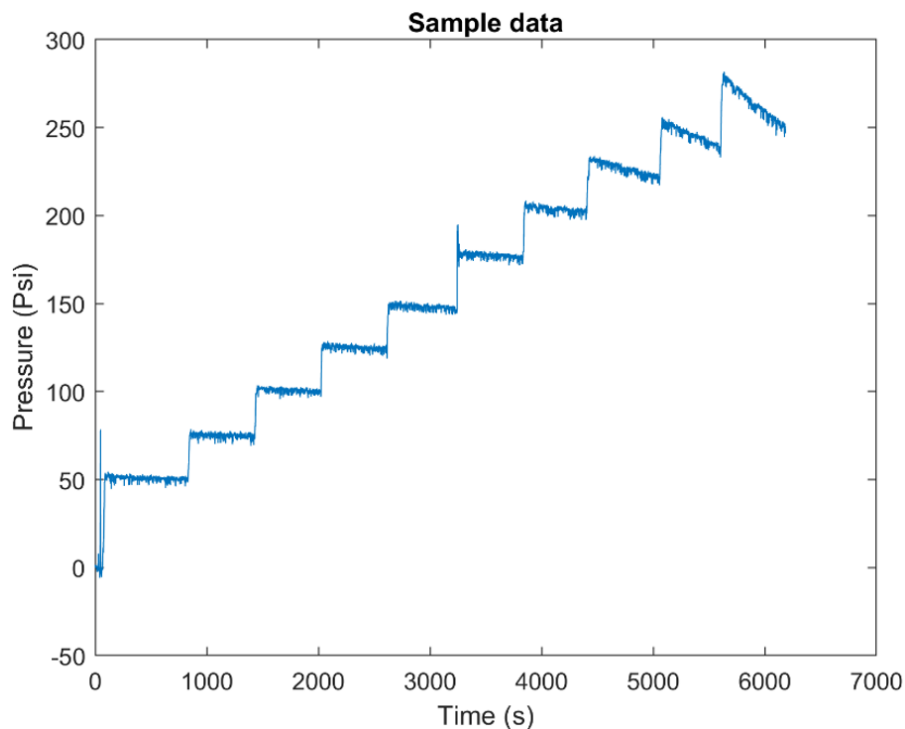


Fig. 5 Pressure decay test on the first sample from coupon 1, coupon-1-1

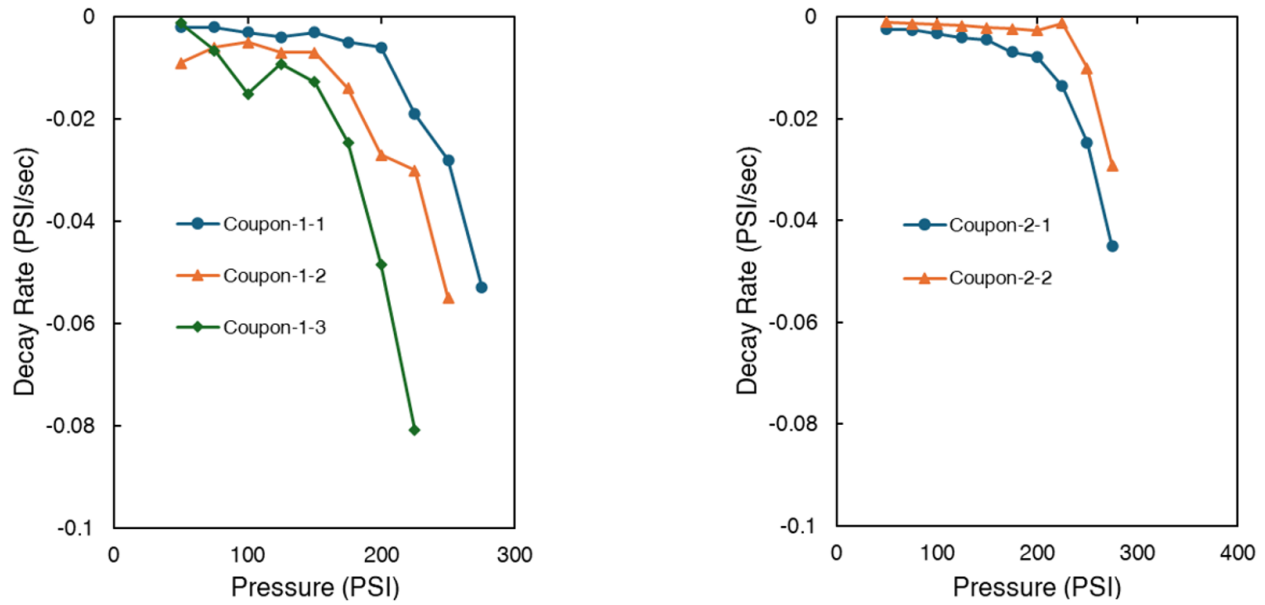


Fig. 6 Pressure decay rate vs. pressure for coupon-1 and coupon-2

point occurs at or above 200 PSI. This increase in the pressure limit could be attributed to increased wall thickness in the second specimen geometry.

Adhesion Characterization of Polymer-metal Interface

Single fiber pullout experiments

In order to characterize the adhesion of the polymer-metal interface, standard single fiber pullout experiments were performed. To better duplicate the actual embed conditions for the fibers, single wire specimens were prepared with wall geometries that were similar to those that were going to be used in real structures, such as polymer heat exchangers (PHXs). Therefore, a single metal fiber was embedded into printed polymer ends that had 1mm thick walls in various polymers, such as PETG, Tritan, and glass fiber infused PETG (**Figure 7**). One end of the specimen was printed as a 25 mm × 20 mm × 5

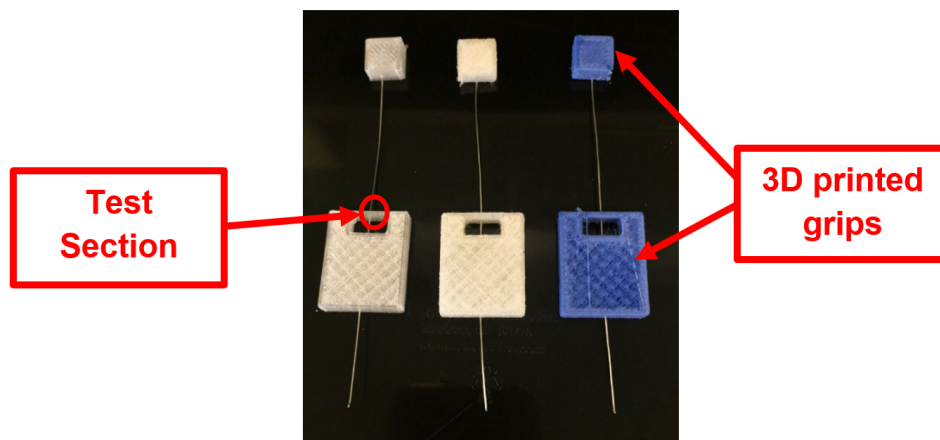


Fig. 7 Single Fiber Pull Out Test specimens with test section consisting of 1 mm thick polymer wall and a gap to cut and release the wire for pullout testing. From left to right are the various polymers we have successfully used to print test specimens: (a) PETG, (b) ABS, and (c) glass fiber infused PETG.

mm grip with a 5 mm gap and a 1 mm wall thickness on one side of the gap, which was chosen so that it mirrors a typical wall thickness used for an end application, such as a heat exchanger. The free-standing wire across the gap is cut to release the resistance from the length embedded into the grip, resulting in a gage length of 25 mm for the remaining free-standing portion of the wire, which has a diameter of 0.4 mm. A 4043 aluminum wire in the annealed condition was used.

The single fiber pullout tests can be performed for a variety of materials using a number of manufacturing techniques. These tests supported the development of a print head that uses both heat and mechanical force to embed the metal fibers and optimize adhesion. Preliminary investigations found very low bond strengths found when the fibers were placed into the print at room temperature, which necessitated improving the processing conditions for embedding the wire. These tests also focused more on PETG as a polymer of interest because of its ease of use and extremely high bond strengths are attractive for many potential applications, such as heat exchangers. The load-displacement results can be seen in **Figure 8** for a specimen that was printed with the wire heated to 175 °C and a nozzle temperature of 238 °C for the glass fiber filled PETG. Note, the maximum load would produce a stress of 135 MPa in the metal wire, which exceeds the strength of the metal wire. Thus, the metal wire begins to draw out plastically before localized tensile failure occurs. Therefore, the performance is not limited by the interfacial strength, but rather the tensile strength of the metal wire.

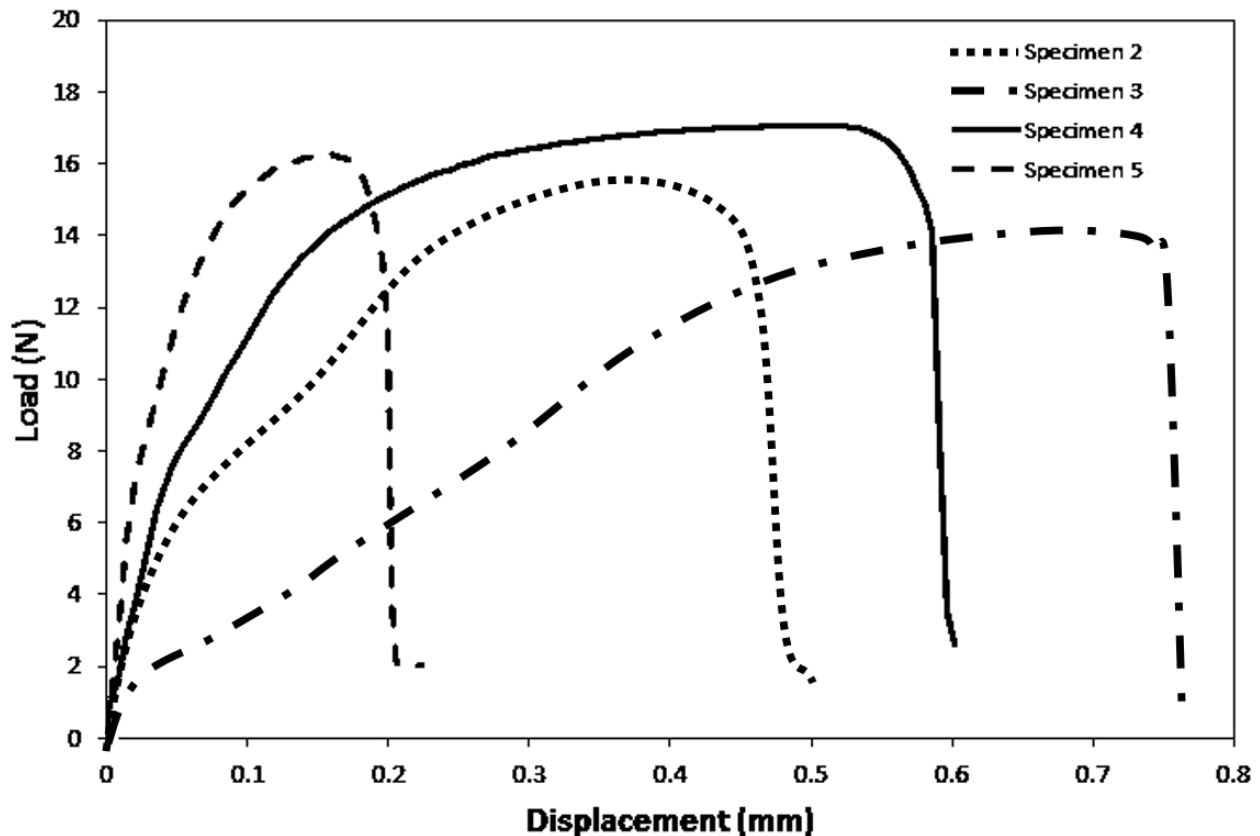


Fig. 8 (left) Load-displacement results for single fiber pullout specimens using metal wires embedded under optimal processing conditions that produced a shear strength that exceeded that need to initiate tensile failure of the metal wire (right).

Thermal characterization of FDM material during processing

A critical aspect of the wire inlay process is the effects of changing over from the FDM print head to the wire inlay head. During that time, the cooling of the FDM material changes the wetting and adhesion of the interface as the material undergoes transition from the melt to the solid phase. To better understand the effects of the state of the FDM material on the strength of the polymer-metal interface, it was necessary to characterize the thermal evolution of the FDM material during processing.

To characterize the thermal evolution of the material, thermocouple measurements were obtained in situ. Results can be seen in **Figure 9** for ABS. The thermal inertia of the thermocouple resulted in a peak temperature 8 seconds after deposition. However, the temperature did plateau at 100 °C, which is near the glass transition temperature of ABS, for another 12

seconds, before undergoing convective cooling. The temperature rapidly decays for another 60 seconds to approximately 40 °C, before reaching nearly steady state conditions associated with the bed temperature of the printer. It is during this 60 second period that the material undergoes phase transformations controlled by the convective cooling rate, which impacts the flow of the polymer at the wire surface, and the subsequent adhesion that develops as the polymer hardens. Therefore, mechanical characterization was conducted, to determine the sensitivity of inlaying the wire at different times on the level of adhesion.

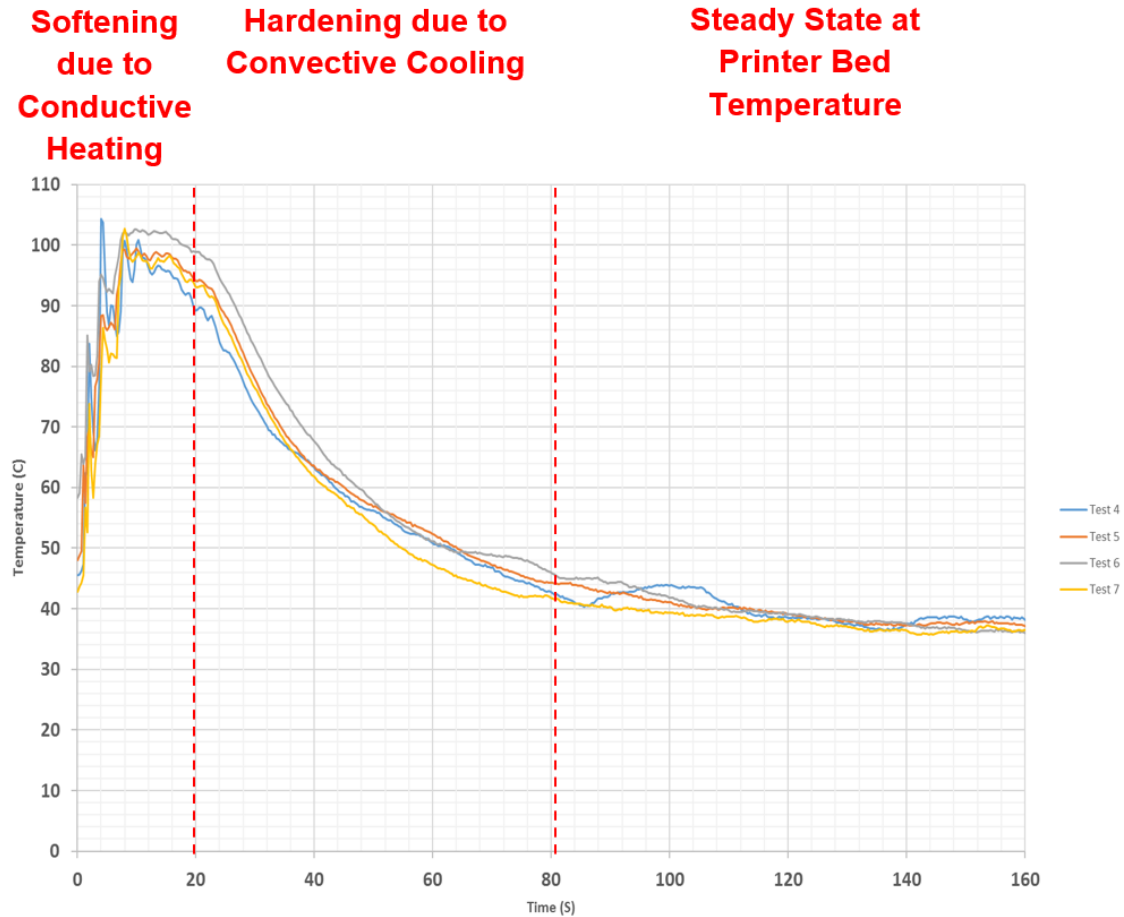


Fig. 9 Thermal evolution of FDM material during the printing process used to determine the time range in which to investigate the effect of inlaying the metal wire on adhesion, where the polymer cools to a steady state temperature of 40 °C with 60 seconds after deposition.

Mechanical characterization of metal wire/polymer support matrix interface for different processing conditions

Representative load-displacement results for the single fiber pullout specimens consisting of the aluminum wire inlayed onto ABS can be seen up until the ultimate load in **Figure 10**. Wire inlay times of 25 sec and 60 sec after the time for the control specimen were also investigated, as well as the use of abrasively polishing the metal wire to remove contaminants and promote adhesion. From these results, it can be seen that the interfacial strength is sufficient to permit the wire to begin to deform plastically, as with the glass fiber-filled PETG. A statistical comparison of the yield and ultimate loads obtained from these curves can be seen **Figure 11**. It can be seen that the polished surface had a slightly higher ultimate loads than the unpolished for all inlay times, with the ultimate load being nearly twice the yield for the polished control specimens. However, the load for 25 s inlay times were 50% lower than the control with no signs of yielding, and the inlay at 50s resulted in a similar reduction relative to 25 s. This is not unexpected, since as the polymer is in a less viscous phase for adhesion, with the reduction being approximately proportional to the temperature at the inlay time relative to the difference between the initial melt temperature and the final bed temperature of the printer.

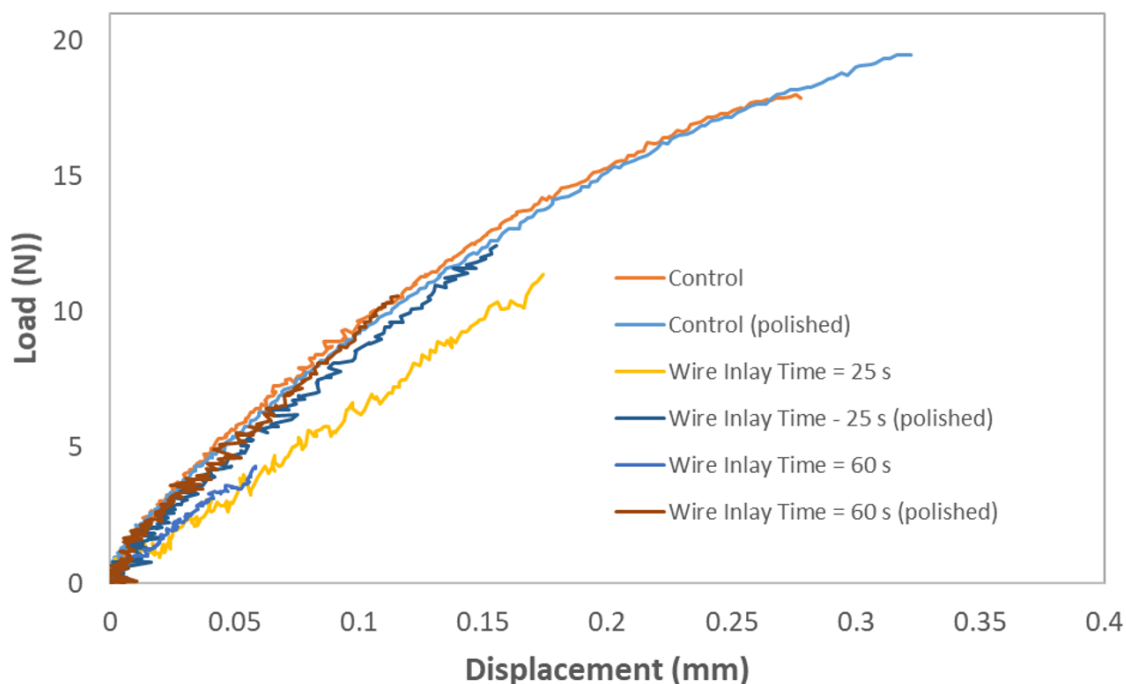


Fig. 10 Representative load-displacement curves for single fiber pullout specimens with polished and unpolished metal wires inlaid at different times relative to the control specimens.

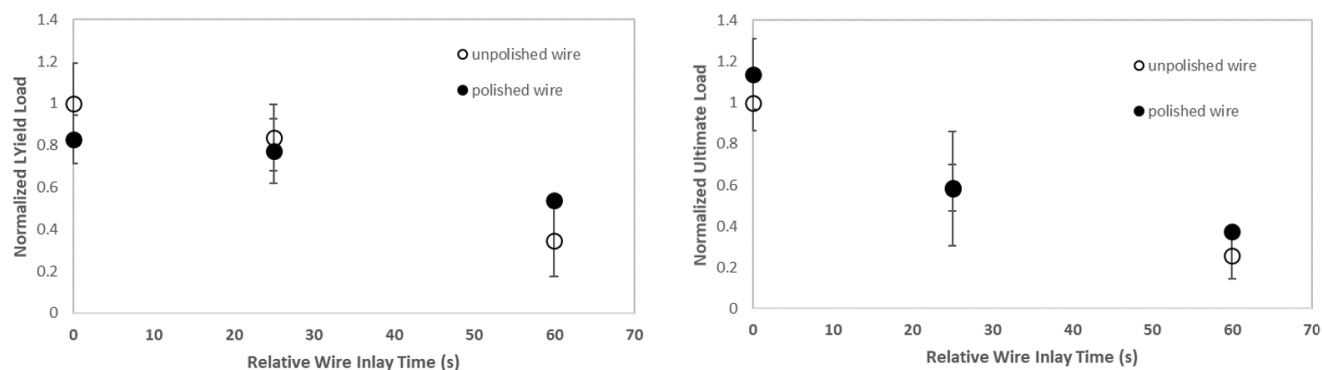


Fig. 11 Statistical comparison of average (left) yield and (right) ultimate loads normalized by the average values of the control specimens, as well as their corresponding standard deviations for single fiber pullout specimens with polished and unpolished wires inlaid at different times relative to control specimens.

Conclusions

The development of 3D printed polymer composite structures with partially free-standing wires embedded in them is affording new opportunities to develop conductive structures that are optimized for lighter weight in a variety of applications, such as heat exchangers. However, there are new challenges in fabricating them to be with enough integrity at the metal wire/polymer matrix interface that they can withstand operating conditions involving pressure differentials that can cause the wires to pullout out and leakage to occur. In this investigation, we have developed a test specimen for characterizing the integrity of the metal wire/polymer matrix interface. The specimen was a small polymer pressure vessel that had multiple partially free-standing metal wires embedded in the walls. These specimens could then be pressurized to determine the loading which the metal wire/polymer matrix interface would fail. Pressures up to 150 psi could be sustained without failure, and up to 200 psi if the wall thickness is increased. Fiber pullout test specimens were also fabricated to determine the interfacial strength. It was determined that optimal conditions could be obtained using heated wires, although the adhesion

was determined to reduce relative to the inlay time for the metal wire, up until the bed temperature of the printer is reached 60 seconds after deposition of the polymer melt. This was attributed to the polymer hardening and not being able to flow sufficiently at the wire surface in order to form good adhesive contact with the wire.

Acknowledgments Financial support of this work by the U.S. Department of Energy, ARPA-E division, under award number DE- AR0000584 is gratefully acknowledged. The views and opinions of authors expressed herein do not state or reflect those of the United States Government or any agency thereof.

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