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Zhang, Wenqiang; Ye, Haitao; Feng, Xiaobin; Zhou, Wenzhao; Cao, Ke; Li, Maoyuan; Fan, Sufeng; Lu, Yang

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Tailoring mechanical properties of P\\textit{\textmu}SL 3D-printed structures via size effect

Wenqiang Zhang\textsuperscript{1,2,8}, Haitao Ye\textsuperscript{1,3,8}, Xiaobin Feng\textsuperscript{4,8}, Wenzhao Zhou\textsuperscript{2}, Ke Cao\textsuperscript{1,5}, Maoyuan Li\textsuperscript{1,6,\ast}, Sufeng Fan\textsuperscript{1,7} and Yang Lu\textsuperscript{1,2,\ast}

1 Department of Mechanical Engineering, City University of Hong Kong, Kowloon, Hong Kong SAR 999077, People’s Republic of China
2 Nano-Manufacturing Laboratory (NML), City University of Hong Kong Shenzhen Research Institute, Shenzhen 518057, People’s Republic of China
3 Department of Mechanical and Energy Engineering, Southern University of Science and Technology, Shenzhen 518055, People’s Republic of China
4 Hubei Key Laboratory of Theory and Application of Advanced Materials Mechanics, Wuhan University of Technology, Wuhan 430070, People’s Republic of China
5 School of Mechano-Electronic Engineering, Xidian University, Xi’an 710071, People’s Republic of China
6 State Key Laboratory of Materials Processing and Die & Mould Technology, School of Materials Science and Engineering, Huazhong University of Science and Technology, Wuhan 430074, People’s Republic of China
7 School of Mechanics and Safety Engineering, Zhengzhou University, Zhengzhou 450001, People’s Republic of China

E-mail: maoyuali@cityu.edu.hk and yanglu@cityu.edu.hk

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Abstract

Projection micro stereolithography (P\\textit{\textmu}SL) has emerged as a powerful three-dimensional (3D) printing technique for manufacturing polymer structures with micron-scale high resolution at high printing speed, which enables the production of customized 3D microlattices with feature sizes down to several microns. However, the mechanical properties of as-printed polymers were not systematically studied at the relevant length scales, especially when the feature sizes step into micron/sub-micron level, limiting its reliable performance prediction in micro/nanolattice and other metamaterial applications. In this work, we demonstrate that P\\textit{\textmu}SL-printed microfibers could become stronger and significantly more ductile with reduced size ranging from 20 \textmu m to 60 \textmu m, showing an obvious size-dependent mechanical behavior, in which the size decreases to 20 \textmu m with a fracture strain up to \sim 100\% and fracture strength up to \sim 100 MPa. Such size effect enables the tailoring of the material strength and stiffness of P\\textit{\textmu}SL-printed microlattices over a broad range, allowing to fabricate the microlattice metamaterials with desired/tunable mechanical properties for various structural and functional applications.

8 These authors contributed equally to the paper.
\ast Authors to whom any correspondence should be addressed.

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1. Introduction

Projection micro stereolithography (PµSL) as an advanced layer-by-layer 3D printing technique provides sub-micron scale high resolution without sacrificing the printing/fabrication speed, which has attracted numerous attentions in many application areas such as electronics, medical, and automotive [1]. The principle of the PµSL is free-radical photopolymerization of liquid photopolymer resin, mainly composed of monomers, crosslinkers, and oligomers. The liquid resin was then cured to covalently crosslinked solid when exposed to UV light. In particular, there is an emerging trend to employ PµSL technologies to fabricate customized bio-devices, micro-electro-mechanical systems, and mechanical metamaterials (such as microlattices) with feature sizes as small as several tens of microns [2–6]. PµSL provided an excellent combination of high printing resolution (0.6 µm–30 µm) and wide printing area (~90 mm × 50 mm) [1] paving paths for these devices and metamaterials. However, there are also growing questions about whether these polymers would behave differently, such as becoming brittle when the size steps into micro/nanoscales. For example, the diameter of 3D printed microneedle tips reaches 20 µm while the claimed fracture strain is 2.2% [7, 8]. Though in most cases, these PµSL printed devices were proved to be safe in their corresponding service environment, the mechanical performance deduced according to their bulk materials is worrying. A deep understanding of micro PµSL printed polymer will accelerate the usage and development of PµSL technologies.

The ‘smaller is stronger’ characteristic, also referred to as one of the ‘size effects’, has been proven in various micro/nanoscale materials, such as metal/alloys, silicon nanowires, diamond, and even ice microfibers et al [9–12]. The size effect can be attributed to many factors/mechanisms, including the minimization of flaws and defects as well as surface effects, etc. In fact, the polymer has been also reported to demonstrate pronounced size-dependent mechanical properties when the dimension decreased to microscale [13–15]. For example, nano acrylic polymer nanowires were reported to have a higher shear modulus ranging from 1.4 GPa to 0.6 GPa remarkably higher than the bulk materials with a modulus of 0.29 GPa [14]. However, only compression tests were conducted in 3D printed spring-shaped polymer micro/nanowires so far. Direct tensile of polymer micro/nanofibers, especially tension in the indoor environment, is less reported for it is difficult to fix the ends of the fiber. In this work, we fabricated PµSL-printed microfibers with integrated clamping ends and the uniaxial tension tests were conducted in the ambient environment. We reported the direct measurement of the mechanical properties of individual fibers and tuned the effective mechanical properties of stretching-dominated octet lattice structures via the size-dependent effect.

2. Preparation of microfiber specimens by PµSL

Microfibers were prepared by high precision PµSL via P130 (BMF Precision Technology Co. Ltd.) with a power intensity of 7.5 µW cm−2 and printing time of 20 s (figure 1(a)). The resin used in this work was acrylic acid resin provided by boston micro fabrication (BMF) Precision Technology Co. Ltd. (Meth) acrylate monomers are the most widely used polymer resins for PµSL. This acrylic resin is brittle with a fracture strain of 3%–5% according to the previous work and experiment data provided by the supplier [7, 16, 17]. Dog bone tensile sample for tensile test comprised two parts: fiber and clamp, as shown in figure 1(b). The fiber was designed with a square cross-section. The width is dominated by the width of UV light and the height is controlled by the gap between the platform and resin surface. All the working part was printed by single exposure. We adopted strategies with low light intensity and a long exposure time of 20 s which makes a balance between curing depth when printing microfiber with a section size of 60 µm and a printing resolution when fabricating the fiber with a section size of 20 µm. To increase the stiffness of the clamps, additional dozens of clamp parts were added layer by layer with a layer-height of 40 µm (figure 1(c)). The post-processing was consistently performed after the tension fixtures were prepared. Then the tensile samples were removed from the platform after 5 min of immersion in alcohol. The as-printed tensile sample was sufficiently post-cured in a curing oven for 25 min and tested 3 d later to ensure stability/repeatability. The as-prepared sample is shown in figure 1(d), consistent with the design schematic diagram in figure 1(c). It should be noted that the length of 3D printing microfiber is measured to be 1500 µm. The inset picture in figure 1(d) is a zoomed-in picture of microfiber. The PµSL printed sample presents a smooth surface which could be a key factor to yield a high strain as it could avoid early-stage local stress concentration.

3. Uniaxial tensile tests of microfibers with different diameters

The tension test of microfiber remains challenging for it is difficult to fix the fiber end [18]. Traditionally, rubber or silver epoxy cladding was employed as a clamping end [19]. Here, dog-bone shape tensile samples with microfiber and clamping ends were fabricated at the same time. Then, the dog bone sample was mounted on a pair of stainless-steel fixtures
Figure 1. Preparation and characterization of PμSL microfiber. (a) The tensile was fabricated by stereolithography, and the working part and clamping part was simultaneously fabricated by a single exposure method. (b) Schematic of the tensile process. (c) Additional clamping sections were designed to increase the stiffness. (d) The SEM image of the fabricated tensile sample.

and stretched as shown in figure 2(a). The tensile test was conducted in Microtest (Gatan) equipped with 20 N load cell with a velocity of 0.2 mm min⁻¹.

Figure 2(b) exhibits the optical snapshot of the tension process of fiber with section sizes of 60 µm and 20 µm. Traditionally, the acrylic polymer was believed to have a fracture strain of about 5%, and the as-printed sample from this resin suffer catastrophic failure [16, 20]. It was interesting to note the micro size fiber is extremely ductile compared with the macro one (Video 1). The fracture strain of fiber with 60 µm was measured to be ∼30%. When the section size decreased by 20 µm, the fracture strain increased to 100% (figure 2(c)). Besides the tensile process, the first frame after the fracture was also presented in figures 2(b) and (c). An obvious resilience was detected in microfiber indicating the ultra-elongation composed of both elastic and plastic deformation.

Figure 3(a) shows the typical tensile curves of the polymer fiber. The tensile deformation of microfiber acrylic resin fiber experienced five stages: elastic deformation, yield, strain softening, cold drawing, and strain hardening. Different stages correspond to the different modes of molecular movements and alignments [21]. As shown in figure 3(a), polymer fibers experience 5% elastic deformation, with a yield stress of about 20 MPa. Followed by a strain-softening stage, the flow stress decreases slightly. When deformation enters the cold drawing stage, a necking occurs in fiber, corresponding to the plateau in the tensile curve [22, 23]. At the cold drawing stage, the necking moves from one side to the other side with a slight increase in stress (Video 2). While in the strain hardening stage, the stress increases with strain. The final strain is up to ∼100% for the polymer microfiber with a section size of 20 µm.

Specifically, as-printed polymer fibers with different cross-section diameters are quantitatively evaluated, and their mechanical properties generally show size-dependent behavior. The initial linear parts of the stress–strain curves (insert in figure 3(a)) show an increasing trend in the modulus as the reduced diameter. More significantly, with the section size decreasing to 20 µm, the fracture strain goes up to ∼100% with corresponding fracture strength up to ∼100 MPa (figure 3(b)). In another word, 3D printed polymer fibers appear to become stronger and significantly more ductile with reduced diameter, showing an obvious size dependence tendency. Figures 3(c) and (d) show the fracture cross-section surfaces of the microfiber with 30 µm and 60 µm, respectively, suggesting different fracture morphologies.

4. Understanding the size effect for microlattice metamaterials

There is a transition from plastic fracture to ductile fracture when the size of acrylic fiber is reduced to tens of microns. Generally, the size-dependent mechanical properties were attributed to the minimization of surface or inner defects. The pre-existing flaw or defect could dramatically undermine the practical strengths of macro-scale materials to several orders of magnitude of the theoretical strengths. And the strength for fracture has an inverse relationship with the size of the defect. The fracture strength will approach the theoretical limit once the defect size decreases to the order of micron/nanometres. The size-dependent behavior of polymer fibers can be partly attributed to the surface effect. Polymer chains typically exhibit higher mobility when they are exposed on the material
Figure 2. *In situ* mechanical test of PµSL printed microfiber. (a) *In situ* test set up for stretching a microfiber with dog-bone shape. Snapshot of the tensile process of PµSL printed microfiber with section size of (b) 20 µm (c) 60 µm. The first frames after fracture are also presented. (The figures (b) and (c) share the same scale bar in figure (c)).

Surface than those within the bulk of the materials [24]. With a smaller/thinner polymer sample, the portion of the surface polymer chains is higher, and thus the overall strength of the sample could be increased (figure 3(e)). Meanwhile, Polarized Raman microspectroscopy in [13] also indicated that the polymer chain in the smaller polymer fiber possessed a greater degree of orientation, of which samples were prepared by the direct laser writing via two-photon polymerization (similar to the forming principle of the PµSL method).

Moreover, previous work on PµSL printed polymer by synchrotron x-ray has detected the nonuniform distribution of defects [25–27]. Generally, the Weibull distribution can also reflect part of the size-dependent mechanical properties (in particular fracture strength and ductility) [28, 29], and it was expressed as:

\[
\ln \left\{ \ln \left[ 1 \left( 1 - P_i \right) \right] \right\} = \ln V + m \ln \sigma_f + m \ln \sigma_o
\]

where \( m \) is the reliability parameter known as the Weibull modulus, \( \sigma_o \) is a scaling parameter, \( V \) is the volume of the tested samples, and \( P_i \) is the fracture probability of materials at given uniaxial stress. In this work, the Weibull modulus is calculated to be 1.5, which is a relatively low value compared with other materials such as SiC, Al₂O₃, and Aluminum [30]. The low Weibull modulus \( m \) reveals that defects are clustered inconsistently rather than uniform distribution.

Meanwhile, there are works reporting fabrication parameters such as the degree of conversion (DOC) may also affect the mechanical properties of a cured polymer [31, 32]. In this work, single exposure was performed to obtain different fiber thicknesses and the DOC difference occurred in the depth.
According to the calculation in [32], the DOC gap in depth of 20 μm and 60 μm is about 5%, leading to a relatively small mechanical difference. As previously described, the as-printed tensile samples were sufficiently post-cured in a curing oven for 25 min and the samples were tested 3 d later to ensure stability/repeatability. Therefore, the overall influence of DOC with sufficient post-processing should be rather limited here. It should be noted that the elasticity and yield stress here are largely insensitive to their size which is similar to the previous study, which are measured by the tensile test [31]. In situ tensile test has proved the ductility of PμSL printed microfiber, and it also proves the bulk brittle polymer could work in a more ductile manner at the microscale, despite that more efforts should be paid to clarify the transition regime of fracture behavior.

Incorporating the size effect of the PμSL printed polymer allows us to obtain microlattice with different mechanical properties yet the same topography and relative density. Two periodic octet microlattices with different strut diameters were designed as illustrated in figures 4(a) and (b). Two parameters were tuned to achieve the same relative density: one is the unit, L, which was 480 μm and 160 μm, and the other is the diameter of the struts, a, which was 60 μm and 20 μm. The printed lattices by PμSL are shown in figures 4(c) and (d). To ensure the sample with the same dimension, the actual truss with struts of 20 μm is designed with $24 \times 24 \times 12$ units while $8 \times 8 \times 4$ units for the truss with struts of 20 μm.

An obvious difference was observed in the two lattices though they have the same relative density (figures 4(e) and (f)). The modulus of the microlattice with 20 μm is measured to be $\sim 87$ MPa about twice compared with that of the lattice with struts diameter of 60 μm ($\sim 43$ MPa). As revealed in the microfiber, the probability of containing fatal flaws in small-size fiber is low. In-situ synchrotron x-ray tomography reveals that these defects have a great influence on the
mechanical properties of the structure. Under compression, these imperfections cause cell rotation and eventually lead to structural damage and the modulus deviation has also been reported [27]. So, the lattice with a 20 \( \mu m \) diameter has higher yield stress and modulus. After the lattices were yielded, the stress of the lattice with 20 \( \mu m \) struts increased with the strain. This may be attributed to the strain hardening phase of the micro-sized struts. While the lattice with a diameter of 60 \( \mu m \) shows typical stress curves of brittle materials. This result reminds us to consider the size of the polymer when designing microlattice metamaterials. More importantly, both the lattice shows different deformation mode compared with the previous work, in which polymer hollow lattice with a wall thickness of 200 \( \mu m \) catastrophically failed [20].

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Figure 4. Tailoring the mechanical properties of the microlattices based on the size effect of struts. (a) Microlattice with struts of 20 \( \mu m \). (b) Microlattice with struts of 60 \( \mu m \). Both A and B are designed with a relative density of 12.7\%. P\( \mu \)SL printed microlattice with struts diameter of (c) 20 \( \mu m \) and (d) 60 \( \mu m \). To ensure the same volume of printed sample, the lattice with struts diameter is designed with a 24 \( \times \) 24 \( \times \) 12 while the lattice with struts diameter is designed with an 8 \( \times \) 8 \( \times \) 4. The load and unload curves of the as-printed sample with struts diameter of (e) 20 \( \mu m \) and (f) 60 \( \mu m \).
5. Conclusion

In this work, we prepared 3D-printed microfibers, as a basic element of microlattice metamaterials, by high precision Projection Micro-Stereolithography. The mechanical properties were systematically investigated by in situ tensile tests. Integrated dog bone tensile samples were fabricated with diameters ranging from 20 µm to 60 µm. Microfiber performs like rubber when the size is decreased to 20 µm with a fracture strain up to ~100% and fracture strength goes to ~100 MPa. The tensile deformation of acrylic microfiber demonstrates five stages: elastic deformation, yield, strain softening, cold drawing, and strain hardening. Such size-dependent mechanical behavior of PµSL-printed acrylate-based resin structures enables the tailoring of the material strength and stiffness of microlattice units over a wide range. The obtained insights also enable the rational fabrication of microlattice scaffolds with desired/programmable mechanical properties for the development of novel micro/nano-lattice mechanical metamaterials.

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ORCID iDs

Ke Cao https://orcid.org/0000-0001-7857-4467
Maoyuan Li https://orcid.org/0000-0002-4944-7762
Yang Lu https://orcid.org/0000-0002-9280-2718

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