Additive Manufacturing and Compression Performance of Micro-/Nano-Structures Based on Two-Photon Polymerization Technology

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Abstract

Two-photon polymerization (TPP) manufacturing technology has a printing accuracy of 100 nm and is a powerful tool for the high-quality and high-precision fabrication of complex micro- and nanoscale structures. However, the effects of the basic material properties and printing process parameters for TPP additive manufacturing on the resulting structures remain unclear, limiting its application in designs with complex performance requirements. Therefore, this study presents detailed printing and testing procedures. For typical specimens, the optimal printing process for IP-Dip, a material in the IP series of photosensitive polymer resins, was investigated, and reference settings for different parameters such as laser power and scanning speed are provided. The obtained parameters were applied to fabricate vertical pillars and lattice structures, and uniaxial compression tests of the specimens were conducted using an in situ SEM/FIB nanomechanical testing system. The compressive modulus of the material is reported, and the compression curves of micro- and nanoscale lattice structures showing layered failure and unloading rebound during compression are presented.

Full Text

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Abstract: Two-photon polymerization (TPP) manufacturing technology has a printing accuracy of 100 nm and is a powerful tool for the high-quality and high-precision fabrication of complex structures at micro- and nano-scales. However, the basic material properties for TPP additive manufacturing and the influence of printing process parameters on structures remain unclear, limiting its application in designs with complex performance requirements. Therefore, this study presents detailed printing and testing procedures. For typical specimens, the optimal printing process of the IP-Dip material in the IP series of photosensitive polymer resins is investigated, and reference settings are provided for different parameters such as laser power and scanning speed. Using the obtained parameters, pillar and lattice structures are fabricated. Uniaxial compression tests of the specimens are carried out with an in-situ SEM/FIB nanomechanical property testing system; the compressive modulus of the material is given, and compression curves showing delamination failure and unloading rebound of micro-/nano-scale lattice structures during compression are presented.

Keywords: two-photon polymerization technology; additive manufacturing; photosensitive polymer resin IP-Dip; compression test; micro-/nano-structure

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Abstract: Two-photon polymerization (TPP) manufacturing technology with 100 nm printing accuracy is a powerful tool for high-quality and high-precision fabrication of complex structures at micro- and nano-scales. However, the basic properties of materials and optimal printing process for TPP additive manufacturing are still not clear, which limits its application in complex performance requirements design. Therefore, this paper studies the optimal printing process of photosensitive polymer resin (IP-Dip) material of typical experimental test samples and gives the setting of different parameters such as laser power and writing speed. The micron-scale pillar and lattice structure were printed, and the uniaxial compression test of the sample was carried out by the SEM/FIB nano-mechanical testing system. The Young' s modulus of the material was given, and it was found that the nano-scale lattice structure had typical delamination failure

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and unloading rebound phenomenon during the compression process.

Key words: two-photon polymerization technology; additive manufacturing; photosensitive polymeric resin IP-Dip; compression test; micro/nano-structure

Additive manufacturing methods can fabricate structures with complex geometries, bringing new opportunities to the development of manufacturing [1] and greatly promoting progress in such fields as lightweight nano-/microstructures, reinforced structural materials, biomedical devices, and microscale sensing and detection devices [2]. At present, additive manufacturing technology has been used to develop many efficient material structures, such as ultralight metallic honeycomb thermal-insulation structures [3], hierarchical structures [4], three-dimensional nanoscaffolds that can serve as functional osteocytes [5], printing for biomimetic microenvironments [6], and chiral metamaterials [7]. Among micro-/nanostructure printing technologies for additive manufacturing, two-photon polymerization technology realizes photosensitive-resin printing with a minimum feature size of 100 nm by ensuring that polymerization is initiated within an extremely small central region through two-photon absorption. This characteristic has fundamentally changed the process for preparing micro-/nanostructures by laser-based material fabrication, improved the final resolution of structures, and enabled high-quality and high-precision fabrication of complex structures [4,7-8]. SONG Xiaoyan et al. [9] reviewed two-photon polymerization (TPP) technology and introduced its principles, development, and applications. MAGGI et al. [5] proposed three-dimensional variable-stiffness nanoscaffolds for culturing bone cells; FRENZEL et al. [7] established acoustic experiments on chiral metamaterials; BAUER et al. [10] obtained, by pyrolysis, high-strength glassy-carbon nanolattices whose strength is second only to that of diamond; NGUYEN et al. [11] reviewed research on the use of TPP in bioengineering; THIEL et al. [12] fabricated three-dimensional submicron photonic crystal structures; QU et al. [8] realized negative-pressure metamaterials under quasi-static conditions; and KOTZ et al. [13] printed three-dimensional fused-silica microstructures with high thermal stability, good chemical and mechanical stability, a resolution of several tens of micrometers, and a surface roughness of 6 nm.

With the widespread application of TPP, the accuracy of the mechanical properties of printed materials is likewise crucial, and the basic properties of the materials and the optimal printing process parameters cannot be neglected [14-15]. Because the designed structures are relatively complex and are also at the micro-/nanoscale, this poses new challenges for mechanics researchers, such as the applicability of traditional analytical theories and the design of specific experiments. DIMITRIADIS et al. [16] conducted nanoindentation experiments on specific structures using atomic force microscopy (AFM). However, the probe region in this method is too small, and damage to the test sample causes structural collapse. At the same time, the indentation depth is limited, and, owing to the surface nonuniformity of polymers or nanocomposites, the surface parameters obtained by indentation cannot represent the properties of the entire

material. LEMMA et al. [17] obtained Young' s modulus, Poisson' s ratio, and the density and viscosity of materials through static and dynamic analyses; experiments further confirmed that exposure power in two-photon lithography is a key parameter that can affect structural stiffness. In a spectral analysis of printing materials, LIU et al. [18] pointed out that IP-Dip generates relatively large shear stresses and plastic deformation at boundaries or in regions of density variation, thereby producing 5%–10% shrinkage. ALBIEZ et al. [19] carried out compression tests on glassy-carbon nanotubes of different sizes with high strength-to-weight ratios, and found that small nanotubes exhibited elastoplastic deformation before the onset of failure. However, such nanolattices were prepared by pyrolysis on the basis of printed polymer microlattices, and do not constitute research on the mechanical properties of the polymer itself.

In summary, the rapid development of additive manufacturing has had a major impact on manufacturing. However, for TPP-oriented additive manufacturing, the basic properties of materials, the performance of lattice structures, and the optimal printing process remain unclear, which limits their application in designs with complex performance requirements. Therefore, based on typical experimental test specimens, this study investigates the optimal printing process for the photosensitive polymer resin IP-Dip and provides settings for different parameters such as laser intensity and scanning speed. Micrometer-scale columns and lattice structures were fabricated, and uniaxial compression tests of specimens were carried out using an in situ SEM/FIB nanomechanical-property testing system. The selection of different parameters and detailed experimental methods are provided, offering a reference for subsequent research in this field.

1 Experimental Methods

All experimental work in this study was conducted in an ISO 7 cleanroom with constant temperature and humidity. The main experimental instruments used are shown in Fig. 1.

Figure 1(a) shows the two-photon polymerization printing system. By focusing the laser beam inside the material, true three-dimensional rapid prototyping can be achieved, with significant advantages and broad application prospects in the fields of photonic crystals, microelectromechanical systems, tissue engineering, and drug delivery. Conventional photopolymerization, i.e., single-photon polymerization, uses ultraviolet light as the excitation light source; polymerization is initiated after the initiator absorbs one photon and becomes excited. Two-photon polymerization uses a near-infrared femtosecond laser as the excitation light source; polymerization is initiated after the initiator simultaneously absorbs two photons and becomes excited. Conventional photopolymerization uses ultraviolet-wavelength lasers (250–400 nm), whose photon energy is high, so polymerization occurs everywhere the light passes. TPP uses lasers with near-infrared wavelengths (600–1 000 nm); near-infrared photons have low energy, linear absorption and Rayleigh scattering are small, and penetration in

the medium is high [9]. By ensuring that polymerization is initiated within an extremely small central region through two-photon absorption, it realizes photosensitive-resin printing with a minimum feature size of 100 nm, and can print complex structures from the macroscale down to the nanoscale.

(a) Two-photon printing system

(b) Plasma cleaner

(c) Scanning electron microscope

(d) In-situ SEM/FIB nanomechanical property testing system (FT-NMT03)

Fig. 1 Schematic diagram of the experimental equipment

The printing material used in this study is IP-Dip photosensitive resin (Fig. 2). This material is designed specifically for two-photon polymerization lithography systems and can print the finest features. It is biocompatible and non-biotoxic, and can be used for optical metamaterials, diffractive optical elements, and micro/nanorobots. Photosensitive resin is also known as a photosensitive polymer or photoresist; it crosslinks and hardens when exposed to ultraviolet light. Because its refractive index ensures ideal focusing, the highest resolution can be achieved. The corresponding objective selected was $63 \times$ NA 1.4. For the substrate, fused silica was selected to match IP-Dip. The right side of Fig. 2 shows a schematic diagram of liquid IP-Dip to be printed, dropped onto a fused silica substrate with dimensions of $(25 \times 25 \times 0.7)$ mm^3.

Fig. 2 Schematic diagram of TPP resin IP-Dip

Before printing the structure on the fused silica substrate, the substrate must be placed in the plasma cleaner shown in Fig. 1(b). Plasma scanning is used to clean the surface and increase the surface roughness so that the structure can adhere better to the printed structure. After the structure is printed and cleaned, the surface must be coated with gold and placed in the scanning electron microscope (SEM) shown in Fig. 1(c) for preliminary observation of its external dimensions and flatness.

Mechanical-property testing was carried out using the in-situ SEM/FIB nanomechanical property testing system shown in Fig. 1(d). Based on micro-electromechanical fabrication technology, this system is equipped with a highly sensitive micro-force sensor probe, redefining the standard for accurate quantitative research in the micro/nanoscale field; its detection accuracy can reach 0.5 μN. By loading the micro-force sensor onto the structure, controlling the magnitude and direction of the applied force in the system settings, and measuring deformation with a position encoder, the compression test required in this study can be completed. The measurement principle of the compression test is to obtain the structural stiffness from the measured displacement (Fig. 3). The deformation during the test includes the substrate (including the SEM fixing pins and the adhesive used to fix the substrate) + the structure to be

tested + the test probe. Calibration is required before the compression test; that is, the probe is pressed directly onto the substrate, and the measured result is taken as the system stiffness. The micro-force sensor selected in this study was a silicon probe with dimensions of 50 μm × 50 μm, capable of measuring forces from −200 000 μN to +200 000 μN. This testing process must be carried out inside the SEM to determine the precise position of the probe and ensure alignment between the structure and the indenter tip.

Labels in Fig. 3: calibration; compression test; k_{probe}; $k_{\text{substrate}}$; $k_{\text{test specimen}}$; probe (silicon); test specimen (IP-Dip printed structure); substrate (quartz glass, adhesive, SEM fixing pins).

Fig. 3 Schematic diagram of the compression test

2 Additive Manufacturing of Micro/Nanostructures

2.1 Parameter Settings

Based on the characteristics of the two-photon polymerization laser printer, several basic parameters must be determined in advance. Before printing, the substrate was cleaned with acetone and isopropanol and dried with an air gun. The substrate was then treated with oxygen plasma to clean and activate it again, giving it stronger adhesion to the structure. The power was set to 80 W, the duration was 5 min, the oxygen flow rate was 5 sccm (1 sccm = 1 mL/min), and the pressure was 0.1 bar (1 bar = 100 kPa). The comparison of printed structures before and after cleaning of the substrate surface is shown in Fig. 4 and Fig. 5; the bottom of the structure without surface treatment cannot adhere well to the substrate.

In this study, a JEOL JSM-6010LA scanning electron microscope was used to observe the structural morphology and monitor the compression-test process in a high-vacuum environment at an accelerating voltage of 5 kV. Because IP-Dip is not conductive, a JEOL JFC-1300 automatic fine sputter coater was used to deposit a thin metal layer on the sample surface by vacuum evaporation. After electron bombardment in the scanning electron microscope, gold atoms can escape, thereby forming secondary-electron or backscattered-electron signals, improving image quality and resolution and clearly revealing the characteristic details of the sample surface. To ensure that the generated sputtering force had the minimum influence on the structure, the current was set to 10 mA, the duration to 100 s (pressure 0.5 mbar), and an approximately 20 nm gold layer was formed on the sample at a distance of 25 mm from the source.

Figure 4 Schematic diagram of the printed structures without oxygen plasma treatment on the substrate surface

Fig. 4 Schematic diagram of the printed structures without oxygen plasma treatment on the substrate surface

Figure 5 Schematic diagram of the printed structures with oxygen plasma

treatment on the substrate surface

Fig. 5 Schematic diagram of the printed structures with oxygen plasma treatment on the substrate surface

Laser power (LP) and exposure time, namely writing speed (WS), are two key parameters in the printing process. Laser power determines the initial region in which free radicals are generated by the initiator, while writing speed affects the number of free radicals generated by the initiator. Lower power can reduce the region in which free radicals are produced, and shorter exposure time can reduce both the number of free radicals generated and their diffusion. By controlling these two factors, the TPP region can be regulated

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. Therefore, trial printing is required, that is, selecting certain LP and WS values to print test specimens. Usually, WS is first set within a certain range, and then LP is varied. If bubbles are produced, this indicates that the power is too high. As shown in Fig. 6, where the horizontal coordinate is writing speed —8 000, 9 000, and 10 000 μm/s from left to right—and the vertical coordinate is light intensity—30, 40, 50, and 60 mW from top to bottom, respectively, a structure consisting of 4×4 pillars and a surface layer covering the pillars was printed. The printing parameters were varied as follows: in the vertical direction, LP increased from 30 mW to 60 mW; in the horizontal direction, WS increased from 8 000 μm/s to 10 000 μm/s. It was found that, if the speed and power were mismatched, shrinkage occurred in the upper right, or pores were generated in the lower left. Meanwhile, Lemma et al.

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pointed out that increasing LP also leads to an increase in strength. Therefore, $LP = 40$ mW and $WS = 10\,000$ μm/s were fixed for subsequent printing. At the same time, the humidity was maintained at 50%, and the temperature was kept unchanged at 20 °C.

The horizontal hatching distance (HD) and the vertical slicing distance (SD) are also important parameters in the printing process; the cutting schematic is shown in Fig. 7. In this study, these two parameters were kept consistent in subsequent printing, both being 100 nm.

Visible labels in Fig. 6: rows: 30 mW, 40 mW, 50 mW, 60 mW; columns: 8 000 μm/s, 9 000 μm/s, 10 000 μm/s.

Figure 6 Structures under different printing parameters

Fig. 6 The structures under different printing parameters

Visible labels in Fig. 7: 3D model → horizontal hatching distance → vertical slicing distance; horizontal hatching distance; vertical slicing distance.

Figure 7 Schematic diagram of horizontal and vertical cutting of the structure during additive manufacturing

Fig. 7 Schematic diagram of horizontal and vertical cutting of the structure during additive manufacturing

2.2 Printing results

After setting the corresponding parameters, cubic structures with side lengths of 2, 4, 6, and 8 μm were printed in this study and measured in SEM (Fig. 8). The error between the printed structures and the design was less than 1%. Therefore, the design dimensions could be used for calculation.

Topology optimization is a highly advanced design method that can fully release the design space and obtain lightweight, ideal structural configurations. In this study, the classic MBB beam in topology optimization was first taken as an example. The topology-optimized configuration was converted into STL format, and printing was performed after parameter setting. Figure 9 shows the printing result of a 60 μm $\times$ 20 μm MBB beam structure. The stair-stepping effect in the figure is caused by the mesh being designed as 60×20, meaning that one mesh cell corresponds to 1 μm. Topology optimization and additive manufacturing

the combination made possible the fabrication of complex configurations —especially microscale structures—and provided support for realizing high-performance structural designs.

Fig. 8 Cubes with side lengths of 2, 4, 6, and 8 μm, and their size measurements in SEM.

Fig. 9 SEM micrograph of the printed MBB beam topology result.

The printed structure of the ultrahigh-strength glassy-carbon nanolattice design created by BAUER et al. [10] exhibited a material strength as high as 3 GPa, equivalent to the theoretical strength of glassy carbon. In this study, their design was used as a reference, and complex lattice honeycomb structures were printed. Fig. 10 shows the printing results for lattice structures with heights of 18, 24, and 30 μm, as well as their measurements in SEM. The error between the printed structures and the designs was less than 1%, demonstrating that the selected parameters are accurate and feasible for structural fabrication.

Fig. 10 SEM diagram of printed lattice structures with heights of 18, 24, and 30 μm, and their size measurements in SEM.

3 Uniaxial Compression Test

3.1 Parameter Settings

An in-situ SEM/FIB nanomechanical performance testing system was used to test the printed structures. First, an appropriate testing probe was selected according to the size and stiffness of the structure, and the system stiffness was

tested. In this study, silicon was selected, with a size of 50 μm × 50 μm. Table 1 shows the system stiffness under different compression displacements.

Fig. 11 Load-displacement curve.

Table 1 System stiffness under different compression displacements (directly pressed on the substrate)

Displacement/μm	0.36	0.48	0.6	0.72	0.84	0.96	1.08	1.2	1.32	1.44	1.56
Stiffness/(N·m^{-1})	26 223	26 782	27 022	27 127	27 515	27 598	27 678	27 772	27 723	27 861	27 901

It can be seen that when the structural height differs, that is, when the compression displacement differs, the system stiffness is also different. Therefore, pre-compression should be performed before testing, and the value should be entered into the system parameter settings. Regarding the selection of the compression speed, LEMMA et al. [17] pointed out that, to prevent viscoelastic materials from bending, the testing speed should be greater than 1 μm/s. Fig. 11 shows the load-displacement curve used to calculate the system stiffness. At this time, the compression displacement was 0.36 μm, and the loading segment from 10% to 70% was selected for stiffness measurement, yielding a system stiffness of 26 233 N/m.

3.2 Test Results

Figure 12 presents a schematic of the uniaxial compression test for a 20 μm × 20 μm × 50 μm pillar. During the test, the system stiffness was first set; the contact surface was then located by preloading at a prescribed speed; finally, compression was performed over the specified compression distance, followed by unloading after continuous loading for 2 s.

Fig. 12 Schematic of the compression test for a 20 μm × 20 μm × 50 μm pillar

Fig. 12 Compression test for a 20 μm × 20 μm × 50 μm pillar

The corresponding load-displacement curve is shown in Fig. 13. Because polypropylene resin has viscoelastic characteristics, stress relaxation appears in the loading curve. However, during the loading stage, the overall response shows a linear trend. Therefore, the Young' s modulus of the material can be obtained by selecting the slope of the 10%-50% portion of the loading stage and using the pillar height and cross-sectional area.

Fig. 13 Load-displacement curve in the compression test of a 20 μm × 20 μm × 50 μm pillar

Fig. 13 Force and displacement curves in the compression test of 20 μm × 20 μm × 50 μm pillar

According to mechanics of materials, the specific calculation of the Young' s modulus is based on the compressive strain of the structure:

$$\varepsilon = \frac{\Delta l}{l} = \frac{\sigma}{E} = \frac{F}{AE} \tag{1}$$

Thus, the Young' s modulus E can be obtained as

$$E = \frac{Fl}{\Delta l \cdot A} = \frac{F}{\Delta l} \cdot \frac{H}{A} = k \cdot \frac{H}{A} \tag{2}$$

where H is the pillar height, A is the cross-sectional area, and k is the stiffness of the compressed structure measured from the slope of the curve within the linear elastic region. The experiment was repeated three times, and the average Young' s modulus was 3.5 GPa. The range specified in bending tests is 0.5–4 GPa[17], indicating that the compression testing method used in this study is accurate.

Compression tests were performed on the lattice structure shown in Fig. 10, and the measured load–displacement curve is shown in Fig. 14. The compression distance was set to 50% of the structural height. It can be clearly observed that the lattice structure underwent layered failure and rebounded after unloading. This microstructure exhibits a significant energy-absorption effect and can be used in engineering applications requiring vibration reduction and energy absorption.

Fig. 14 Load–displacement curve in the compression test of the lattice structure

Fig. 14 Force and displacement curves in the compression test of lattice structure

4 Conclusion

This study presents a detailed procedure for printing compression-test specimens by two-photon polymerization additive manufacturing, together with the selection of relevant parameters. Before fabrication, the substrate must be plasma-cleaned to ensure good contact between the structure and the substrate, while maintaining constant temperature and humidity throughout the experiment. Because the resolution of the structure depends on the laser power LP and scanning speed WS, $LP = 40$ mW and $WS = 10\,000$ μm/s were selected for subsequent printing. Meanwhile, the transverse distance HD between two adjacent printed layers of the structure and the thickness SD of each sliced layer were both set to a fixed value of 100 nm.

Using the above parameters, cubes, topology specimens, and complex lattice honeycomb structures were fabricated; the error between the printed structures and the designed structures was less than 1%. During the uniaxial compression test, a nanomechanical testing system was used to preload the test structure

to obtain the system stiffness, after which the test speed and distance were specified to perform the uniaxial compression experiment. From the obtained load–displacement curve, the Young' s modulus of the 20 μm × 20 μm × 50 μm pillar was calculated to be 3.5 GPa. The compression curve also revealed the layered failure of the micro/nanoscale lattice structure during compression and its rebound after unloading. The proposed printing and testing procedure can provide a reference for subsequent research in this field.

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Note: Figure translations are in progress. See original paper for figures.