EXPERIMENTAL AND THEORETICAL STUDY ON ELASTIC MODULI OF POROUS PDMS WITH DIFFERENT POROSITIES AT DIFFERENT TEMPERATURES

Chen Huang[1, a], Chengfeng Fang[1, b], Xiaoliang Zhou[2], Zuguang Bian[3,*]

[1]College of Civil Engineering and Architecture, Zhejiang University, Hangzhou, 310058, China, [a]chenhuang@zju.edu.cn, [b]fcf2012@zju.edu.cn

[2]Institute of Solid Mechanics, Beihang University (BUAA), Beijing, 100191, China, zhouxiiiaoliang@163.com

[3]Ningbo Institute of Technology, Zhejiang University, Ningbo, 315100, China, bzg@nit.net.cn

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ABSTRACT

In recent years, polydimethylsiloxane (PDMS) has been extensively used in many technological areas, such as flexible electronics and microfluidic systems. When it comes to flexible electronics, porous PDMS are necessary in clinical application, only few studies, however, have been reported on the mechanical properties of porous PDMS. In present paper, distilled water was selected as porogen to fabricate porous PDMS, and samples with different porosities were produced by altering the ratio of water to PDMS prepolymer. We experimentally measured the elastic moduli of PDMS with different porosity in a uniform temperature field, and found that the elastic modulus was decreases as its porosity increases. We also measured the elastic moduli of porous PDMS at different temperatures, ranging from 0℃ to 40℃, experimental results indicate that the elastic modulus increases as its temperature raises. Meanwhile, a two-scale asymptotic homogenization method (AHM), incorporated with a finite element method (FEM), was employed to estimate the elastic moduli of porous PDMS. The results coincide with those measured in experiments, which verifies both the validity of our measurement data and the applicability of homogenization theory in predicting the physical properties of porous composites. In our following stage, more mechanical characteristics of porous PDMS will be investigated, which are essential parameters in studying the mechanical performance of epidermal flexible electronics.

1 INTRODUCTUION

PDMS is a synthetic polymer which is common material of flexible electronic substrate and possesses lots of advantages, such as good insulation, excellent optical characteristic and high elasticity. Since the concept of flexible electronics was proposed [1], it developed quickly and has been used in many products, such as hemispherical electronic eye camera [2], flexible inorganic light-emitting diodes [3] and epidermal electronics [4]. Although there are different products, flexible

* Corresponding author.

electronic devices are usually composed of several parts, including electronic component, flexible substrate, interconnect and bonding adhesive [5]. Considering of the flexibility and deformability, polymer is suitable as substrate, like polyimide (PI), PDMS, polyethylene terephthalate (PET), etc.

Recently, PDMS has been extensively used as substrate in clinical flexible electronics due to its biocompatibility and stability. When it is employed in a stretchable epidermal sensor for long-term monitoring, the PDMS must have open pores within it to assure the sweat penetration. Mechanical characteristics of porous PDMS have fundamental effect on the stretchability of electronic devices, few studies on which, however, have been reported. H.Y. Wang et al developed porous PDMS membranes by hydrosilylation curing [6]. The pores are formed by the reaction between the SiH and OH groups. O. Dufaud et al prepared porous PDMS particles on the basis of the chemical crosslinking of water in PDMS emulsion droplets in a hermostated water vessel [7]. M. Juchniewicz et al fabricated porous PDMS by the emulsion polymerization technique [8]. Distilled water, acting as the porogen, was mixed into PDMS by a stirrer. After cured at 120℃, porous PDMS with open pores could be obtained. Some researchers used porogen leaching technique to fabricate porous PDMS [9-10]. A porogen such as sugar or salt particles were added into PDMS prepolymer and mixed before curing the mixture, and then followed by washing away the porogen in water to leave porous material.

Actually, many researchers have studied the factors that affect the mechanical characteristics of PDMS. However, almost all the researches focus on pure PDMS. K. Khanafer et al studied the effects of mixing ratio of prepolymer and curing agent on the elastic moduli of PDMS [11]. Their results indicate that elastic moduli increase as the mixing ratios increase up to 9:1, and then the elastic moduli start to decrease as the mixing ratios continue to increase. M. Liu et al researched the mechanical strength of PDMS membranes with different thickness. They demonstrated that the elastic moduli of the PDMS membranes are thickness dependent and the transition from bulk behavior to dimension dependent is predicted to occur at a membrane thickness of about 200 μm [12]. M. Liu et al further reported that the heating temperature influences the mechanical properties of PDMS [13]. They pointed out that for low heating temperatures, the mechanical characterizations are independent of heating time, and higher heating temperatures produce lower mechanical strength. F. Schneider et al analyzed the dependence of the elastic moduli on the thinner concentration, temperature and strain rate [14].They observed a linear correlation between the elastic moduli and temperature and a slightly increasing elastic moduli as a function of the strain rate. I.D. Johnston investigated the variation in the mechanical properties of PDMS with curing temperature, over the range from 25℃ to 200℃ [15]. They found a linear relationship between the elastic moduli and the curing temperature.

In this study, we used the emulsion polymerization technique to fabricate porous PDMS. By adjusting the ratio of water to PDMS prepolymer, porous PDMS with different porosities can be obtained. A tensile test was then followed to gain stress-strain curve. Considering that the mechanical characteristics of PDMS are sensitive to its temperature, we also measured the elastic moduli of porous PDMS at different temperature ranging from 0℃ to 40℃. Besides, the mechanical properties of porous PDMS were theoretically evaluated by the homogenization theory, which is frequently employed in multi-scale analysis, especially in predicting the macro effective physical parameters of composite materials. In our study, a two-scale AHM in corporate with a finite element model was established to predict the effective elastic moduli of porous PDMS with different porosities, which were then compared with the experimental results.

2 EXPERIMENTAL

2.1 Fabrication

PDMS (Sylgard®184) was purchased from Dow Corning, including silicon elastomer base and silicon elastomer curing agent. The ratio of base to curing agent was 10:1, distilled water was added into the prepolymer. By controlling the proportion of water to PDMS prepolymer, samples with different porosity can be fabricated. In this study, silicon elastomer base and water were combined at various weight ratios designated as A, B, C, D and E, corresponding to 8:0, 8:4, 8:5, 8:6 and 8:7.

A mechanical stirrer was then used to mix distilled water with PDMS prepolymer in a high speed, after that the mixture was degassed in a vacuum desiccator for 2 h. After removing the bubbles generated when stirring, the prepared mixtures were poured into a mold and cured in an oven at 120°C, which was far higher than the boiling point of water. Therefore, the water evaporated very quickly and porous PDMS with open pores can be obtained.

2.2 Tensile Test

The tensile test was performed according to Rubber, vulcanized or thermoplastic—Determination of tensile stress-strain properties (ISO 37:2011), in which the ISO 37:2011 Type 4 standard was used. All the PDMS samples were cut into dumbbell shaped structures (Figure 1) by a cutter and divided into five groups, including pure PDMS (A) and porous PDMS with different porosity (B, C, D and E). The outer dimension of the test pieces are 35x6x1 mm^3, whereas the length and width of the test section measure 10x2 mm^2. Tensile testing of the samples was carried out on a universal testing machine (Zwick/Roell Z020) according to ISO 37:2011. The crosshead velocity was 15 mm/min. The samples were tested in an incubator with the temperature varies from 0°C to 40°C. In order to obtain a representative test results, five samples were tested for each group.

Figure 1: Photograph of test samples, upper: pure PDMS, down: porous PDMS.

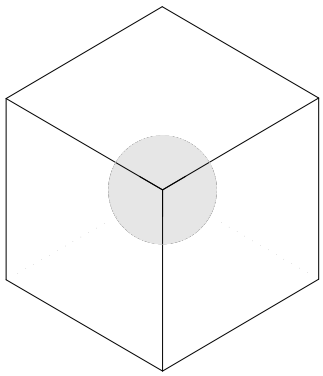


Figure 2: Unit cell of theoretical analysis

3 THEORETICAL ANALYSIS

Homogenization theory was widely used in many areas of physics and engineering, especially analysing the properties of composite materials, such as computing the heat transfer coefficient in

porous media. Materials with a periodic structure can be represented by a base cell which is the smallest repetitive unit of material. The base cell contains all the properties of materials, and the evaluation of the effective physical parameters may be carried out by analysing the base cell (Figure 2) alone [16]. According to the principle of virtual work, the virtual displacement equation of the base cell can be written as:

$$\int_{\Omega^\varepsilon} E_{ijkl} \frac{\partial u_k^\varepsilon}{\partial x_l^\varepsilon} \frac{\partial v_i}{\partial x_j} \mathrm{d}\Omega = \int_{\Omega^\varepsilon} f_i^\varepsilon v_i \mathrm{d}\Omega + \int_{\Gamma_t} t_i v_i \mathrm{d}\Gamma + \int_{S^\varepsilon} p_i^\varepsilon v_i \mathrm{d}S \tag{1}$$

Here $\varepsilon=x/y$ ($0 < \varepsilon << 1$) is introduced, which is a small parameter that connects the macroscopic coordinates x and the microscopic coordinates y. Ω^ε is the solid part of the base cell, E_{ijkl} is elastic modulus of solid part in the base cell, u is the displacement boundary and v is arbitrary virtual displacement which satisfies the boundary conditions. f is the body force, t is the tractions and p is the tractions in the hole.

Then, using the double-scale asymptotic expansion, u^ε can be written as:

$$u^\varepsilon = u^0(x,y) + \varepsilon u^1(x,y) + \varepsilon^2 u^2(x,y) + \ldots \tag{2}$$

Now, using the perturbation method and considering the periodicity of the unit cell, three control equations can be obtained:

$$\int_{\Omega^\varepsilon} E_{ijkl} \frac{\partial u_k^0}{\partial \mathrm{y}_1} \frac{\partial v_i}{\partial y_j} \mathrm{d}\Omega = \frac{1}{|Y|} \int_\Omega \int_\Lambda E_{ijkl} \frac{\partial u_k^0}{\partial \mathrm{y}_1} \frac{\partial v_i}{\partial y_j} \mathrm{d}Y \mathrm{d}\Omega = 0 \tag{3}$$

$$\frac{1}{|Y|} \int_\Omega \int_\Lambda E_{ijkl} \left[\left(\frac{\partial u_k^0}{\partial x_1} + \frac{\partial u_k^1}{\partial \mathrm{y}_1} \right) \frac{\partial v_i}{\partial y_j} + \frac{\partial u_k^0}{\partial \mathrm{y}_1} \frac{\partial v_i}{\partial x_j} \right] \mathrm{d}Y \mathrm{d}\Omega = \frac{1}{|Y|} \int_\Omega \int_S p_i v_i \mathrm{d}S \mathrm{d}\Omega \tag{4}$$

$$\frac{1}{|Y|} \int_\Omega \int_\Lambda E_{ijkl} \left[\left(\frac{\partial u_k^0}{\partial x_1} + \frac{\partial u_k^1}{\partial \mathrm{y}_1} \right) \frac{\partial v_i}{\partial x_j} + \left(\frac{\partial u_k^1}{\partial x_1} + \frac{\partial u_k^2}{\partial \mathrm{y}_l} \right) \frac{\partial v_i}{\partial y_j} \right] \mathrm{d}Y \mathrm{d}\Omega = \frac{1}{|Y|} \int_\Omega \int_\Lambda f_i v_i \mathrm{d}Y \mathrm{d}\Omega + \int_{\Gamma_t} t_i v_i \mathrm{d}\Gamma \tag{5}$$

By solving the control equations, the linear elasticity problem can be written as:

$$\int_Y E_{ijpq} \frac{\partial \chi_p^{\mathrm{kl}}}{\partial y_q} \frac{\partial v_i(y)}{\partial y_j} \mathrm{d}Y = \int_Y E_{ijpq} \frac{\partial v_i(y)}{\partial y_j} \mathrm{d}Y \tag{6}$$

Here χ_p^{kl} is characteristic displacement which is the solutions of the base cell. As the focus of research on composite materials, the homogenized elastic modulus E^{H} can be denoted:

$$E_{ijkl}^{\mathrm{H}} = \frac{1}{|Y|} \int_\Lambda \left(E_{ijkl} - E_{ijpm} \frac{\chi_p^{kl}(x,y)}{\partial y_m} \right) \mathrm{d}Y \tag{7}$$

Where E_{ijkl}^{H} represents homogenized elastic constants and Y is the volume of the base cell.

As shown above, the effective elastic modulus can be obtained by solving Eqs. (6) and (7) with the help of finite element method. However, different from general problems in elasticity, people need to write corresponding program to solve the unit cell problem above, which is tedious for complicated structure. Therefore, the homogenized elastic modulus E_{ijkl}^{H} is written in another form [17]:

$$E_{ijkl}^{\mathrm{H}} = \frac{1}{|Y|}\int_Y \left(\varepsilon^{0(ij)} - \varepsilon^{*(ij)}\right)^{\mathrm{T}} E\left(\varepsilon^{0(kl)} - \varepsilon^{*(kl)}\right)\mathrm{d}Y \tag{8}$$

The equation is denoted in matrix notation, whose physical meaning is strain energy of the base cell. Here, E is constitutive matrix of the material, ε^0 is unit strain field which is achieved by applying unit displacement χ^0, ε^* is characteristic strain field corresponding to characteristic displacement χ^{kl}.

The finite element formulation of Eq. (6), which is constrained with periodic boundary conditions, can be written as:

$$K\chi^{kl} = f^{kl} \tag{9}$$

Where

$$K = \int_Y B^{\mathrm{T}} EB\mathrm{d}Y \tag{10}$$

$$f^{kl} = \int_Y B^{\mathrm{T}} E\varepsilon^{0(kl)}\mathrm{d}Y \tag{11}$$

Here B is the finite element strain-displacement matrix. The nodal force vector can also be denoted as:

$$\begin{aligned} f^{kl} &= \int_Y B^T E\varepsilon^{0(kl)}\mathrm{d}Y \\ &= \int_Y B^T EB\chi^{0(kl)}\mathrm{d}Y \\ &= \int_Y B^T EB\mathrm{d}Y\,\chi^{0(kl)} \\ &= K\chi^{0(kl)} \end{aligned} \tag{12}$$

The homogenized elastic modulus E_{ijkl}^{H} can be transformed as:

$$\begin{aligned} E_{ijkl}^{\mathrm{H}} &= \tfrac{1}{|Y|}\int_Y E_{pqrs}(\varepsilon_{pq}^{0(ij)} - \varepsilon_{pq}^{*(ij)})(\varepsilon_{rs}^{0(\mathrm{kl})} - \varepsilon_{pq}^{*(\mathrm{kl})})\mathrm{d}Y \\ &= \tfrac{1}{|Y|}(\chi^{0(ij)} - \chi^{(ij)})^{\mathrm{T}} K(\chi^{0(kl)} - \chi^{(kl)}) \\ &= \tfrac{1}{|Y|}(\chi^{0(ij)} - \chi^{(ij)})^{\mathrm{T}}(f^{(kl)} - f^{*(kl)}) \end{aligned} \tag{13}$$

Commercial finite element software was used to calculate the homogenized elastic modulus, the calculation process can be summarized as:

i. Ansys was used to create the model and separate grid, the results of group A were used as the elastic moduli of the solid part in the base cell, and the Poisson ratio is 0.48.
ii. Unit displacement field χ^0 which is equivalent to unit strain field ε^0 was applied to each node. For example:

$$\chi_{node}^{0(11)} = \begin{Bmatrix} x \\ 0 \\ 0 \end{Bmatrix} \quad \chi_{node}^{0(12)} = \begin{Bmatrix} y \\ 0 \\ 0 \end{Bmatrix} \tag{14}$$

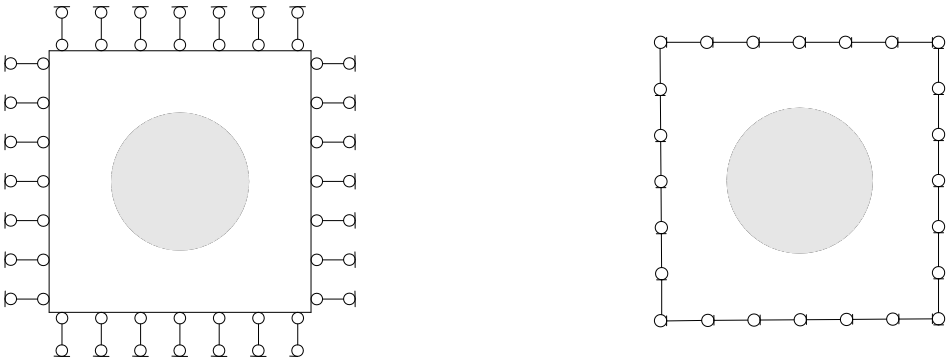
iii. Running the static analysis to obtain nodal force vector f^{kl}

iv. Applying nodal force vector f^{kl} to the model with periodic boundary conditions, running the static analysis to get characteristic displacement χ^{kl}.

v. Applying characteristic displacement χ^{kl} to the model to obtain $f^{*(kl)}$, and calculate homogenized elastic modulus E_{ijkl}^{H} by Eq. (13).

vi. Calculating the homogenized elastic modulus E^{H} according to the constitutive matrix.

The method above is more convenient compared to traditional AHM. Ansys was used as a calculation tool, which can do the entire work, and effective elastic constants can be obtained directly from the output of Ansys. In process iv, periodic boundary condition [18] was achieved by restricting the edges of the base cell, which is shown in Figure 3.



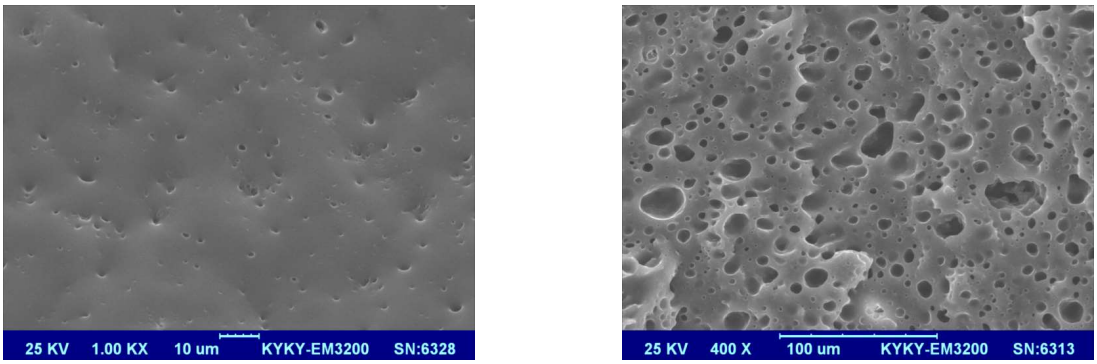
(a) Normal deformation (b) shear deformation

Figure 3: Periodic boundary conditions

4 RESULTS AND DISCUSSION

4.1 Experimental Results

Figure 4 shows the scanning electron microscope (SEM) images of air-side surface and the cross-section side of porous PDMS. Since the porosity of a porous medium at a point is equal to the average value of the areal porosity at that point [19]. The porosities of PDMS were studied by Image-Pro Plus (IPP), which is an image analysis software. Table 1 shows the average pore size and porosities of different ratio of PDMS. It was found that porosity rises with the ratio of water increases; however, the change in the ratio of water has little effect on the pore size.



(a) SEM image of air-side surface (b) SEM image of cross section

Figure 4: SEM images of porous PDMS.

Group	Average pore size (μm)	Porosity
A (8:0)	–	–
B (8:4)	2.96	13.17%
C (8:5)	3.03	16.08%
D (8:6)	3.73	20.25%
E (8:7)	3.21	22.13%

Table 1: Porosity of different ratio of PDMS

Five samples were tested for each group, and the stress-strain curve was determined by averaging the test data of each group. Figure 5 presents the stress-strain curves of different porosities of PDMS at 20 ℃. Experimental results show that stress-strain curves of porous PDMS retains linear until its strain exceeds 40%, which is consistent to other researchers' results [14-15]. The elastic modulus was therefore determined from the slope of stress-strain curve within strain less than 40%. F. Schneider et al [14] mentioned that the wider end section of the test pieces has a significant influence on the measurement of strain in the test section. They also demonstrated that a correction factor can be calculated for strain below 40%, which is dependent on the geometry of the test piece. Ansys was used to simulate the tensile process of test piece. A correction factor 1.2 was obtained by using their method. All the results after corrected are listed in Table 2, from which we can find the elastic modulus of porous PDMS decreases as its porosity increases. It's also found that the elastic modulus of porous PDMS increases as its temperature rises.

4.2 Theoretical Results

Since the unit cell was assumed isotropic, only two load cases were applied to the model to get E_{1111} and E_{1212}. Then, according to the constitutive matrix of material, homogenized elastic moduli E^H can be obtained, which were listed in Table 3. At the same time, the experimental results and theoretical results were presented on Figure 6 to compare them intuitively, in which X axis represents elastic modulus by experiment and Y axis represents elastic modulus by AHM. For

excellent predictions, the theoretical value would equal the experimental value and all data points would sit on the straight, diagonal reference line. It was found that the results of AHM are lower than the experimental results; however, for most points, the variance between two methods wasn't high, which is no more than 15%. As shown in Figure 6, the results by AHM are close to the results by experiment, which confirms the effectiveness of AHM in predicting the properties of porous materials.

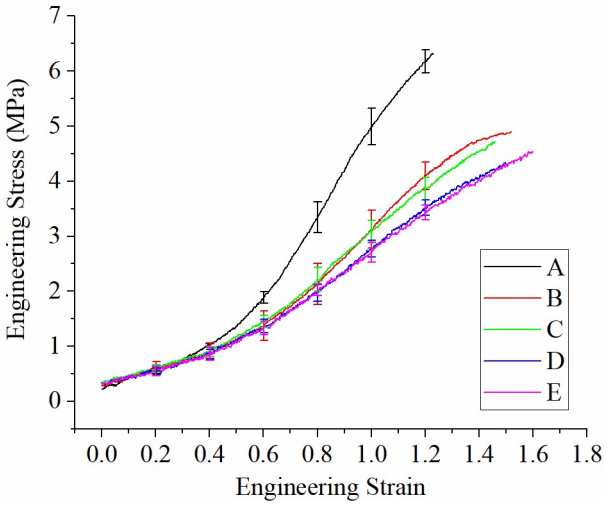


Figure 5: Stress-strain curve of PDMS with different porosities at 20 ℃

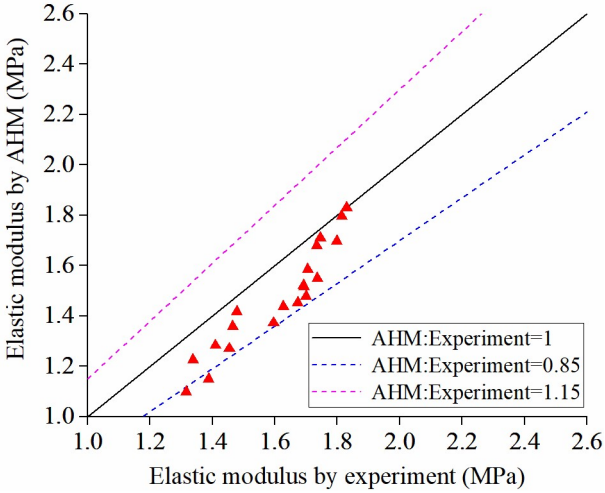


Figure 6: Comparison of elastic modulus by experiment and AHM

Temperature \ Sample	A	B	C	D	E
0℃	1.796±0.010	1.465±0.015	1.454±0.020	1.388±0.018	1.316±0.018
10℃	2.003±0.013	1.694±0.013	1.478±0.012	1.410±0.033	1.338±0.015
20℃	2.242±0.015	1.798±0.016	1.705±0.012	1.627±0.015	1.596±0.017
30℃	2.375±0.009	1.814±0.008	1.734±0.017	1.692±0.008	1.673±0.009
40℃	2.417±0.017	1.830±0.020	1.746±0.031	1.736±0.008	1.700±0.012

Table 2: Experimental elastic moduli of PDMS at different temperatures (MPa)

Temperature \ Sample	B	C	D	E
0℃	1.360	1.271	1.151	1.100
10℃	1.517	1.417	1.284	1.227
20℃	1.697	1.586	1.437	1.373
30℃	1.798	1.680	1.522	1.454
40℃	1.830	1.710	1.549	1.480

Table 3: Theoretical elastic moduli of porous PDMS at different temperatures (MPa)

Considering the effect of pore size and porosity on the mechanical properties, we calculated the elastic moduli of PDMS with different pore sizes by using AHM. It was found that the elastic modulus is almost the same with the pore size changes, which indicates that the pore size has little effect on elastic modulus when the porosity is constant. The elastic moduli of PDMS with different porosities were also calculated and an equation which can predict the properties of Porous PDMS with different porosities was proposed. The equation is denoted as:

$$E^H = (1.2\theta^2 - 2\theta + 1)E \tag{15}$$

Here E^H is the homogenized elastic modulus, θ is the porosity of porous PDMS and E is the elastic moduli of PDMS of the solid part.

5 CONCLUSION

In this paper, the emulsion polymerization technique was used to fabricate porous PDMS. A tensile testing was performed to obtain the material properties of PDMS with different porosities. It was found that the elastic modulus of porous PDMS decreases as its porosity increases.Since the mechanical characteristics of PDMS change with temperature, the elastic moduli of porous PDMS at different temperature ranging from 0℃ to 40℃ were measured, from which we found the elastic modulus of porous PDMS increases as its temperature rises.

After measuring the mechanical properties of PDMS with experiment, we also analyzed porous PDMS theoretically by homogenization theory. A two-scale asymptotic homogenization method was set up to predict the elastic moduli of porous PDMS with the help of the finite element method. It was found that the predicted results are close to the results of experiment, which verifies the effectiveness of AHM in analyzing the properties of porous material. Then the asymptotic homogenization method was used to analyze the elastic moduli of PDMS with different porosities and pore sizes. It was found that pore size has little effect on elastic modulus, and an equation was put forward to predict the mechanical characteristics of PDMS with different porosities.

Through our experimental analysis on elastic moduli of PDMS with different porosities, we hope to provide data for the design of flexible electronic devices based on porous PDMS. The measured results can also afford theoretical support for the optimization of clinical flexible electronics. At the same time, the effectiveness of asymptotic homogenization in analyzing the properties of porous PDMS was verified, which can contribute to predicting the mechanical characteristics of porous materials.

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