Static and Dynamic Mechanical Characterization of Polydimethylsiloxane (PDMS) under Uniaxial Tensile Loading

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Abstract. The present study is based on performing mechanical characterization of polydimethylsiloxane (PDMS) under tensile loading. PDMS samples are developed by mixing prepolymer and curing agent with 10:1 ratio. Mechanical characterization of the PDMS samples is carried out using static and dynamic mechanical testing methods. In particular, the material is characterized under monotonic loading, different strain rates, and force-controlled cyclic loading. The monotonic loading tests are performed at different grip tightening levels to optimize gripping that avoids slipping and reaches failure point. The strain rate dependency and cyclic loading tests are performed to study the viscoelastic behaviour of the material. Strain rate dependency tests are carried out at the strain rates of $6.7 \times 10^{-3}$ s$^{-1}$, $3.2 \times 10^{-2}$ s$^{-1}$, and $6.2 \times 10^{-2}$ s$^{-1}$. The hysteresis tests are performed at the force rates of 0.1 – 2 N/s. The obtained results show that fixing the testing specimen made of soft hyperviscoelastic material such as PDMS is challenging and can significantly affect its stress-strain behaviour and mechanical properties. A higher strain rate increases the stiffness of the material due to the viscoelastic nature of the material. Observing the stress-strain curves, with respect to the lowest strain rate case, the curve for $6.2 \times 10^{-2}$ s$^{-1}$ strain rate diverges at 40% strain compared to $3.2 \times 10^{-2}$ s$^{-1}$ strain rate curve that diverges at 80% strain. The material shows a nonlinear hysteresis behaviour and similar energy dissipation for the considered force-controlled cyclic loading cases.

1. Introduction

Polydimethylsiloxane (PDMS) is a high-performance most widely used silicone polymer due to its unique mechanical, physical and chemical properties. As an elastomer, PDMS is used in various applications including microfluidics, biomedical, cosmetics and soft robotics. Recently, PDMS has gained huge attention in the development of soft and flexible sensors that are useful to many aerospace and mechanical applications. Kamat et al. [1] developed a PDMS-graphene cantilever-form airflow sensor with excellent sensitivity and repeatability. Montazerian et al. [2] mixed chopped carbon fibers with PDMS to develop piezoresistive composite yarns. Chen et al. [3] developed stretchable strain sensor using PDMS with CNTs (carbon nanotubes) in a sandwich-like form as PDMS/CNTs/PDMS. Kou et al. [4] fabricated a pressure sensor using PDMS and graphene. Development of such sensing soft materials could be useful to fabricate smart and flexible flapping wings for small robotic flyers known as micro aerial vehicles (MAVs) [5,6]. Hence, it is important to study the mechanical behavior of PDMS carefully to develop accurate material models for designing purposes. However, it is a
challenging task due to soft and hyperviscoelastic nature of the material. Some studies have considered the mechanical characterization aspect of PDMS but only a few did it systematically, under tensile mode and characterized the material for static and dynamic mechanical properties. Liu et al. [7] investigated tensile mechanical properties of PDMS membranes of different thicknesses. Authors reported that Young’s modulus and tensile strength depend on the thickness of PDMS films due to shear stress developed with the cross-linked chains during the spin coating process. Liu et al. [8] studied the effect of heating temperature and time on mechanical properties. This study showed that the tensile strength starts to decrease at a temperature higher than 200°C. It was also observed that the heating time does not affect the tensile strength for low-temperature heating. Mata et al. [9] prepared the PDMS samples with different weight percentages (wt%) of cross-linker with prepolymer. They found that the tensile strength increases up to 14.3 wt% and decreases for higher ones, i.e., 21.4 wt% and 42.9 wt%. Khanafer et al. [10] studied the effect of mixing ratio and strain rate on the elastic modulus. The authors reported that the elastic modulus increases with mixing ratio up to 9:1. It was also observed that the strain rate increases the modulus. This effect is more significant for the lower mixing ratio of 6:1 as compared to 9:1. Nunes [11] characterized PDMS in shear mode and measured large deformations using digital image correlation technique. The authors discovered the nonlinear behaviour of shear strain, while other studies reported linear shear stress-strain. Kim et al. [12] also studied the effect of the mixing ratio on the stress-strain behaviour under tensile mode. It was reported that the stress softening occurs during the first unloading cycle and decreases with the mixing ratio (5:1 to 15:1). There are a few studies that carried out dynamic mechanical characterization at different frequencies. Placet and Delobelle [13] reported that the storage and loss moduli increase with the frequency (10^{-2} - 10^{5} Hz). However, Lin et al. [14] reported an increase in storage modulus for 0.1 to 100 Hz, but the loss modulus starts to decrease after 1 Hz.

It has been understood that the proper mechanical characterization and properties of PDMS material are not yet established and still in exploring state. The material has to be characterized systematically by considering proper testing methods and precautions. Therefore, the present study was aimed at performing systematic mechanical characterization of PDMS under tensile loading using static and dynamic mechanical testing methods.

2. Materials and Methods

Given the soft and hyperviscoelastic nature of PDMS, the present study explored a systematic mechanical characterization process. The study employs appropriate testing equipment including low-capacity high-accuracy load cell and screw-controlled rubber-coated lightweight tensile grips. The present study also paid considerable attention to the level of grip tightening of the soft material samples. The material is characterized under monotonic loading, different strain rates and cyclic loading to investigate the hyperelastic as well as viscoelastic characteristics of the material. This section describes the preparation of the PDMS material and the characterization process used for investigating its mechanical properties.

2.1. Material preparation

The PDMS prepolymer and the curing agent (Sylgard 184 manufactured by Dow Corning Corporation) were procured from Sigma-Aldrich. Figure 1 shows the procedure used for making PDMS samples for characterization. The base polymer (prepolymer) and the curing agent are mixed with the ratio of 10:1, as the same proportion is considered in many other studies. The curing is done in an oven at 100°C for 35 min based on the instructions from the maker. The circular shape samples are cured in the petri dish after placing them at a horizontal surface inside the oven. The testing samples are cut using a surgical cutter based on the testing standard used for a particular characterization.
2.2. Static mechanical characterization

It is a well-known fact that testing a soft material such as PDMS is a complicated task, especially in tensile mode. Gripping the specimen at both ends should be done carefully to avoid unwanted initial stress due to compression from the grip surfaces. This stress would cause deformations that would affect the actual initial stress-strain behaviour of the material. At the same time, the gripping should be enough to make the sample fail during testing. Therefore, selecting the grip type and the gripping force is critical to perform tensile testing of PDMS material.

The static mechanical characterization is carried out to perform two studies; one is simple monotonic loading to observe the stress-strain behaviour, and another is done with different testing speeds to observe the effect of strain rate. These tests are performed using Tinius Olsen H10-KT universal testing machine (UTM). Due to the soft nature of the material, 250 N load cell is used to measure force values, and the samples are clamped at both ends using manual screw-controlled rubber-coated light-weight grips to appropriately fix the specimen and control the specimen tightening (see Figure 2). The dimension of the testing specimen and testing parameters are based on ASTM D-412. The specimen dimensions are: gauge length 25 mm, breadth 6 mm, and thickness 1 mm.

**Figure 1.** Procedure for making PDMS samples.

**Figure 2.** Illustration of components of the setup for testing PDMS using UTM, (a) load cell and grips, (b) grips and the failed sample.
2.3. Dynamic mechanical characterization
The dynamic mechanical characterization is performed using a dynamic mechanical analyzer (DMA (MetraVib'100) shown in Figure 3). The tests are performed under force-controlled cyclic mode to study the viscoelastic behaviour of the PDMS material. Testing specimen and parameters are decided using the instruction manual provided by the machine maker. The specimen are fixed at both ends using suitable grips for the tensile testing. The relation which is used to find dimensions of the testing sample is

\[ b > \frac{2tl}{t+l} \]

where, \( l \) is the length, \( b \) is the breadth, and \( t \) is the thickness.

![Dynamic mechanical analyzer with the illustration of the testing setup.](image)

3. Results and Discussion

This section presents the results obtained using UTM and DMA. The obtained results are discussed to study the stress-strain behaviour, strain rate dependency, and energy dissipation capability of the PDMS material.

3.1. Stress-strain behaviour under monotonic loading
We started the monotonic tensile testing of the material with less tightening and increased it until the sample stopped slipping from the grips and reached its failure point. The initial tightening level where the sample did not slip and failed is considered as the medium or less tightening. The full tightening level is considered when the sample is fully tightened by rotating the screw-controlled tightening valve to the maximum. Figure 4 shows two stress-strain tests conducted at the strain rate of \( 6.7 \times 10^{-3} \text{ s}^{-1} \) (crosshead speed of 10 mm/min), one is for medium tightening, and another is for full tightening. Note that the sample failed in both of the cases. It can be seen that tightening a sample at different forces changes the stress-strain behaviour of the material. The material shows less initial stiffness for full tightening in comparison to the one with less tightening level. This can be attributed to the fact that due to higher tightening level, the material will be under deformation before applying tensile force during testing. Basically, the compression due to grips leads to a large desired axial stretch, which is constrained at the surface (no-slip). Hence, local compressive axial stress develops in the grip which would lead to an initial compression in the gauge region. Now as the load is applied, additional stress is experienced by the material, which sees an apparent increase in strain for the same stress. This leads to an apparent loss of stiffness and reduction of strength. In terms of the mechanical properties, the
The respective values of Young’s modulus, tensile strength, and failure strain for full tighten condition are 0.89 MPa, 3.79 MPa, 177.6%, and for medium tighten condition are 1.8 MPa, 7.73 MPa, 141.4%. These are significant differences as far as consistency in mechanical properties is concerned. Hence, the selection or rather designing of grips as well as fixing of samples must be done very carefully to achieve actual stress-strain behaviour and mechanical properties of soft materials such as PDMS.

3.2. Effect of strain rate

The strain rate tests are performed to observe the strain rate dependency of PDMS. The selection of the range of strain rates or crosshead speeds is done on the basis of ASTM D-412. The standard recommends a crosshead speed of 5 mm/min to 500 mm/min for various rubbers and elastomers. In the present study, to study the effect of varying strain rate, three testing speeds are considered, i.e., 10 mm/min, 50 mm/min and 100 mm/min to comply with the ASTM standard and testing speed capability of the UTM machine. The corresponding strain rates based on the gauge length of 25 mm are $6.7 \times 10^{-3}$ s$^{-1}$, $3.2 \times 10^{-2}$ s$^{-1}$, and $6.2 \times 10^{-2}$ s$^{-1}$. Figure 5 shows the results for the considered strain rate cases. The comparison is shown only up to 80% strain level considering that the materials generally work under 80% of strain. Under this strain limit, it can be seen that the material shows negligible strain rate dependency until 40% strain. The strain rate of $6.2 \times 10^{-2}$ s$^{-1}$ diverges at around 40% strain, whereas the other two low strain rate cases are overlapping till the considered 80% strain limit. From these results, it can be concluded that strain rate dependency or viscoelastic nature of the material depends on the strain rate and the strain limit. The increase in strain rate increases the stiffness of the material as it increases the slope of the stress-strain curve. The similar observation has been observed in literature at the testing speed of 5 mm/min and 500 mm/min for PDMS samples made using 9:1 and 6:1 mixing ratio [10]. Another observation is that the stress-strain curve for the higher strain rate case diverges earlier than that with the lower strain rate curve.

Figure 4. Stress-strain behaviour under monotonic loading at two different tightening levels, sample failed in both cases.
3.3. Viscoelastic behaviour via cyclic loading

The cyclic loading tests are performed to estimate the energy dissipation capacity of the PDMS material. The tests are performed in force-controlled mode at 0.1 N/s, 0.5 N/s, 1 N/s and 2 N/s. The length, breadth and thickness of the samples are 4 mm, 4 mm, 2.32 mm, respectively. These dimensions are calculated using the dimension selection equation given in Section 2.3. The results from the cyclic tests are shown in Figure 6. It can be seen that the material shows hysteresis behaviour as the unloading does not follow the loading path. It indicates that the material is viscoelastic. However, the energy dissipation capacity is low due to less area of the hysteresis loop, for all the force rate cases. It can also be observed that the stress-strain curves are nonlinear that indicate the nonlinear viscoelastic behaviour of the PDMS material. Comparing the loading rates, for high force rate loading cycles, the hysteresis loop approaches consistency as the material follow the same path for all the cycles of loading and unloading. Another observation is that the energy dissipation capacity decreases with the increase in force rate but with a minimal difference.
Figure 6. Results from the cyclic loading tests performed using dynamic mechanical analyser, (a) 0.1 N/s, (b) 0.5 N/s, (c) 1 N/s, (d) 2 N/s.

4. Conclusions

The present study provides interesting insights on the hyperviscoelastic nature of soft material PDMS subjected to static and dynamic mechanical loadings in the tensile mode. It is observed that the grip tightening can alter the actual stress-strain behaviour and mechanical properties of the material. Increasing strain rate increases the stiffness of the material owing to its viscoelastic nature. The strain rate tests also showed that the stress-strain curve subjected to a higher strain rate diverges significantly earlier than those with the lower strain rate. Cyclic tests showed the nonlinear hysteresis nature of the material. The hysteresis behaviour happens to be more consistent at a high force rate cycle than the lower ones. However, there was no significant difference in the energy dissipation capacity of the material.

The present study employed appropriate testing equipment and processes to characterize the material with a systematic approach as it is important for a better characterization and modelling of PDMS. The study provides new observations and results that could be helpful to establish systematic mechanical characterization methods for soft materials.

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