



Test method

Contact-free thermal expansion measurement of very soft elastomers using digital image correlation

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ABSTRACT

The coefficient of thermal expansion (CTE) is a critical material property that represents the dimensional stability of a material under thermal loading. For soft materials such as elastomers, accurate measurement of the CTE is challenging due to the severe local deformation of the specimen in contact with the measuring apparatus. In this paper, a contact-free CTE measurement method is introduced for soft polymers using three-dimensional digital image correlation. The accuracy of the method was validated using various reference materials. The CTEs of extremely soft silicone elastomers were successfully measured with high precision, where the specimens were suspended in order to ensure free expansion of the compliant and adherent materials. Moreover, the high-throughput capability of the method was demonstrated through simultaneously evaluating multiple specimens. For polydimethylsiloxane (PDMS), the effects of the mixing ratio and curing temperature on the CTE and fractional free volume (FFV) of the polymer network were investigated, where the correlation between the CTE and FFV was quantitatively verified. The proposed method is expected to satisfy the growing demands for accurate CTE measurement of soft materials.

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1. Introduction

The coefficient of thermal expansion (CTE) is an important material property that indicates the dimensional stability of a material under temperature variations. Because elastomers are frequently subjected to thermal loading, the CTE of an elastomer has been a key consideration in their various applications. The high temperature sensitivity of elastomers, typically polydimethylsiloxane (PDMS), has been exploited in thermally actuated devices [1–4] and thermal sensors [5–9], where the device performance is determined by the CTE of the elastomeric media. The thermal shrinkage of an elastomeric substrate after curing at higher temperatures generates periodic wrinkles on the surface of thin film, which are utilized in optical engineering [10–12], stretchable electronics [13,14] and bioengineering [15,16]. The CTE is used to tune the surface topography [17–20], calculate the thermal stress [15,21] and measure the elastic modulus [22–24] of the thin films.

In contrast, the high CTE of elastomers often causes thermal problems that degrade the fabrication quality, thermal reliability and integrity of the accompanying systems, including soft lithography [25–27] and elastomeric joint sealing systems applied in various areas from microfluidic devices [28,29] to space shuttles [30–32]. Therefore, accurate CTE values for elastomers are essential as crucial design parameters for elastomer-based systems.

Despite the increasing importance of the CTE of elastomers, accurate measurements and systematic studies have been limited due to the lack of appropriate methodology. For example, while the effects of curing PDMS have been well identified for other material properties such as the elastic modulus [33–37], permeability [38–40] and degree of swelling [39,40], there have been no relevant studies presented for the CTE. The conventional methods used to measure the CTE such as the strain gauge, dilatometer and thermomechanical analysis (TMA) are contact-based methodologies that have difficulties in testing soft materials. Because the specimen becomes severely distorted at the local contact area with hard fixtures such as steel grips or quartz probes, the precision of the measurement is significantly lowered and the CTE values become unreliable. Furthermore, the CTE measurement requires a longer period of time compared with other typical mechanical

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properties, which adds one more problem to be solved.

In order to overcome the experimental limitations, this study adopts a digital image correlation (DIC) technique as a non-contact strain measurement method. DIC is a powerful tool for analyzing various types of deformations using a pattern tracking algorithm based on digital images, which is suitable for measuring planar and surface deformations under various loading conditions [41–44]. In recent years, DIC has been used to measure the CTEs of polymeric films [45–48] and tubular steel specimens [49]. Technical progress has been established for measuring small thermal strains [48,49] and optical strategies [48,50]. However, these works cannot be directly applied to elastomers because the elastomer specimen fails to achieve free expansion for the following reasons. First, if the elastomer sample is self-standing on a heating stage as in the previous studies, the compliant sample will adhere to the stage as a result of its high van der Waals adhesion. Second, the speckle pattern sprayed on the entire surface of a specimen constrains the expansion as well, particularly due to the large thermal expansion and softness of the elastomers. In addition, the previous methods are restricted to a paradigm of “one at a time” measurement, which is insufficient for exploring the thermal expansion properties of polymers with various formulations.

In this study, a non-contact CTE measurement method is introduced for extremely soft elastomers through employing real-time 3D DIC. In order to ensure the free expansion of the compliant materials, the specimens are suspended and a two-point strain measurement was adopted. The accuracy, precision and high-throughput capability of the proposed method are demonstrated. The proposed method is then applied to crosslinked silicone elastomers with wide modulus variations. The influences of the mixing ratio and curing temperature on the CTE of PDMS were investigated, where the fractional free volume (FFV) of the PDMS was also measured as a primary source of its macroscopic thermal expansion. The correlation between the CTE and FFV was quantified for the two curing parameters of PDMS.

2. CTE measurement by DIC technique

2.1. Experimental setup

A schematic representation of the setup is presented in Fig. 1. A 3D DIC system composed of a pair of CCD cameras is placed in front of a heating system in order that the DIC system can observe the thermal deformation of a specimen through the glass window of the heating chamber. The precise strain measurement was conducted using a commercial DIC system (ARAMIS, GOM mbH, Germany). Two charge coupled device (CCD) cameras with a resolution of 2448×2050 pixels were calibrated with a camera angle of 23.9° and a base distance of 180 mm. The calibrated 3D space, or measuring volume, was $70 \times 60 \times 40 \text{ mm}^3$ with its center located at a distance of 425 mm from the lens of the image sensors. The maximum measurement error was guaranteed to be less than $1 \mu\text{m}$ within the volume. For the heating system, a forced convection chamber (3119-606, INSTRON, USA) and a digital temperature controller (Eurotherm 3208, INSTRON, USA) were combined. At the center of the chamber, a custom fixture frame was used to hold the sample vertically with the glass grips at the chamber top and the sample facing downwards. The fixture was composed of two parallel glass plates of 1 mm thickness, which prevented the specimen from fluttering due to air disturbances. Between the shield glass plates, two smaller glass pieces of 2 mm thickness were used as grips. The grips also functioned as spacers that provided a 4 mm gap for free expansion of the specimens.

2.2. Thermal strain measurement

In order to obtain free expansion of an elastomer specimen, a two-point strain measurement method was employed that requires minimal markers. First, the reference image of each specimen was obtained at the initial temperature, where two gauge points were assigned with a square subset of 25×25 pixels. The stereovision system measured the 3D positions of the two gauge points with respect to the origin point of calibration (x_0, y_0, z_0): and (x_1, y_1, z_1)

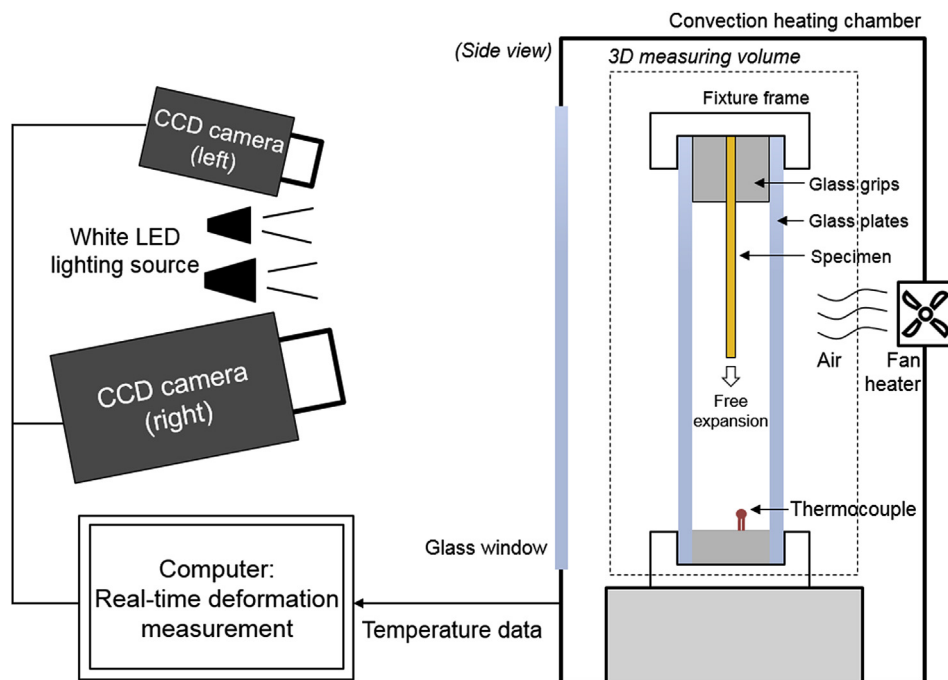


Fig. 1. A schematic of the experimental setup (side view).

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