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Test Method

# Simultaneous measurement of temperature-dependent refractive index and depth-resolved thermal deformation fields inside polymers

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ABSTRACT

Based on phase-contrast spectral optical coherence tomography, a method of simultaneously measuring the temperature-dependent refractive index and depth-resolved thermal deformation field inside polymers is proposed. The interference spectra are acquired before and after the change in the polymer temperature, and the geometrical and optical thickness variations of the polymer are decoded from the spectra. The temperature-induced refractive index change is subsequently estimated using the measured thicknesses variations. With the estimated refractive index change and the phase difference map decoded from the spectra, the depth-resolved thermal deformation field inside the polymer can be evaluated. To validate the method, silicone rubber and epoxy resin films were measured while their temperatures were increased from 50 °C to 100 °C. The measured linear thermal expansion coefficients of the silicone rubber and epoxy resin films were $3.74 \times 10^{-4}$ and $5.85 \times 10^{-5}$, respectively, which are close to the existing recommended data. The advantages of the method are that the depth-resolved thermal deformation field inside the polymers can be measured in real time while eliminating the errors caused by changes in both the polymer refractive index and the medium temperature, as well as system vibration.

## 1. Introduction

Thermal expansion is an important property of polymer materials and structures, especially in high precision engineering applications [1-2]. Many optical methods have been used for investigating thermal expansion, for example, electronic speckle pattern interferometry (ESPI) [3], digital image correlation (DIC) [4], and fiber Bragg grating (FBG) sensors [5]. Among these methods, phase-contrast spectral optical coherence tomography (PC-SOCT) shows promise in measuring the depth-resolved thermal deformation fields inside translucent materials with high sensitivity [6].

PC-SOCT is a combination of phase contrast techniques and spectral domain optical coherence tomography (OCT), which acquires the interference spectra before and after the polymer deformation and evaluates the depth-resolved deformation field from the phase difference of the spectra. Because the spectrum can be captured by the PC-SOCT system within only one shot, the method is available for studying the dynamic deformation inside polymers. In addition, nanometer deformation can also be measured, as the phase difference is sensitive to the optical path difference (OPD) between the sample and reference arms [6-9]. Although conventional PC-SOCT has already become a popular method for observing mechanical load induced polymer deformations, it cannot be well applied to polymer deformations under thermal loads. As the OPD is the product of the refractive index and distance, the temperature-dependent refractive index of the polymer should be predetermined while evaluating its depth-resolved thermal deformations from the spectra [10-12]. However, a non-simultaneous measurement of the temperature-dependent refractive index and depth-resolved thermal deformation will inevitably produce errors as it is a complex measuring process.

Using OCT to measure the geometrical and optical thicknesses of an object is an effective method to obtain the object's refractive index [13]. However, the conventional OCT has poor OPD sensitivity, which is not available for measuring temperature-induced refractive variations. In this paper, temperature-induced nanometer geometrical and optical thickness variations of the polymer were acquired using PC-SOCT for estimating the refractive index variations while evaluating the depth-resolved thermal deformations. The proposed method removes the error caused by the medium temperature change and the system vibration, providing a practical method for polymer testing. In Section 2, the system configuration, the experimental procedure, and the principle of the method are introduced. In Section 3, the measured results of a silicone rubber film and an epoxy resin film are presented and analyzed. The conclusions are then presented.

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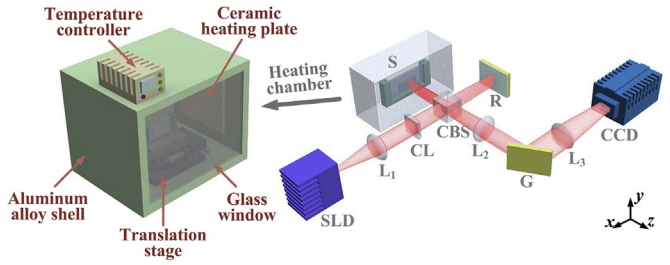


**Fig. 1.** Configuration of the PC-SOCT system, with labels; SLD: Super luminescent diode; $L_1$-$L_3$: lens; CL: cylindrical lens; S: sample; R: reference plane; CBS: cube beam splitter; G: grating; and CCD: charge coupled device.

## 2. Methodology

### 2.1. System configuration and experimental procedure

The system configuration of a PC-SOCT is shown in Fig. 1. The light from a super luminescent diode SLD (Superlum Diodes Ltd., SLD-37-HP2, with a bandwidth of $\Delta\lambda = 50$ nm centered at $\lambda_c = 840$ nm and an output power of 10 mW) is collimated by a lens $L_1$ and focused by a cylindrical lens CL for illuminating a cross-section in the *y*-*z* plane inside the sample S. The backscattered light of S interferes with the light reflected from the reference plane R through a cube beam splitter CBS. After being collimated by a lens $L_2$ and diffracted by a grating G, the interference light forms a spectrum and the image is formed on a CCD camera (Allied Vision Technologies, Manta G-145B, 1388 × 1038 pixels, 12 bits) with a lens $L_3$. The focal lengths of $L_1$-$L_3$, and CL were 60 mm, 150 mm, 300 mm, and 150 mm, respectively; with a grating G of 1200 lines/mm. The heating chamber was composed of a temperature controller, a double layered aluminum alloy shell, a glass window, two symmetrically placed ceramic heating plates, and a set of translation stages. The inner temperature of the chamber was controlled from 30 °C to 150 °C with a precision of ± 1 °C. The measured samples were silicone rubber (Dow Corning 732) and epoxy resin (Bisphenol-A epoxy resin E51) films. When making a film sample, the sample and its substrate surfaces should be accurately flat and in contact with one another. Because the epoxy resin is transparent, the epoxy resin film sample needs to be seeded with $TiO_2$ particles (0.4 μm average diameter) to increase the scattering. The experimental procedure was as follows: 1) the sample was mounted in the heating chamber; 2) the temperature was set to 120 °C; and 3) the interference spectra was captured by the CCD camera continuously with an exposure time of 60 ms and a delay time of 1 s. Photographs of the system, heating chamber, and a film sample are shown in Fig. 2.

### 2.2. Principle of the method

The cross-section of a test sample is shown in Fig. 3(a) where $z_R$ is the depth of the reference plane; and, $z_1$ and $z_2$ are the depths of the front and rear surfaces of the film. Since the optical path length Λ, between R and S, can be obtained from the frequency $f_k$ of the spectrum along its wavenumber *k* axis, [6]

$$\Lambda = \pi f_k, \tag{1}$$

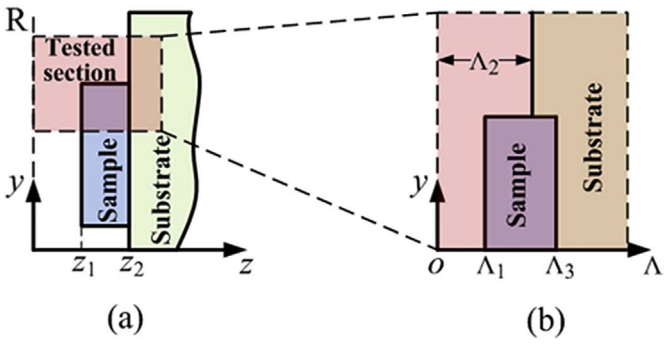


**Fig. 3.** Schematic of the tested section in the (a) *y*-*z* plane and (b) *y*-Λ plane.

then the tested section was estimated from the amplitude-frequency characteristics of the spectrum as shown in Fig. 3(b). Since the refractive indexes of the film and the medium are different, dislocation was observed in the *y*-Λ plane. From the estimated values of $\Lambda_1$, $\Lambda_2$, and $\Lambda_3$, the thickness $t_s$ and the refractive index $n_s$ of the sample can be written as [13]

$$t_s = \frac{\Lambda_2 - \Lambda_1}{n_m}, \tag{2}$$

$$n_s = \frac{(\Lambda_3 - \Lambda_1)n_m}{\Lambda_2 - \Lambda_1}, \tag{3}$$

where $n_m$ is the refractive index of the medium. The resolution of the measured Λ was $\delta\Lambda = \lambda_c^2/\Delta\lambda = 14$ μm, therefore, the resolution of the measured thickness was 14 μm; and the resolution of the measured refractive index was 0.014 for $n_m = 1$ and $t_s = 1$ mm.

The variation in the optical path length ΔΛ was estimated from the phase change ΔΠ of the spectra, before and after the sample deformation as [6]

$$\Delta\Lambda = \frac{\lambda_c}{4\pi}\Delta\Pi. \tag{4}$$

A phase change of 2π corresponds to an optical path length change of $\lambda_c/2$. Therefore, nanometer scale variations of sample deformations can be observed. It is noted that the ΔΠ obtained from the spectra is wrapped and need to be unwrapped before the ΔΛ estimation. If the sample is deformed due to a change in its temperature Δ*T*, the variations of $\Lambda_1$, $\Lambda_2$, and $\Lambda_3$ are

$$\begin{cases} \Delta\Lambda_1 = (z_1 - z_R)\Delta n_m + \Delta z_1 n_m + \Delta\Lambda_0 \\ \Delta\Lambda_2 = (z_2 - z_R)\Delta n_m + \Delta z_2 n_m + \Delta\Lambda_0, \\ \Delta\Lambda_3 = t_s\Delta n_s + \Delta t_s n_s + \Delta\Lambda_1 \end{cases} \tag{5}$$

where $\Delta n_m$ and $\Delta n_s$ are the refractive index variations of the medium and the sample, respectively; $\Delta z_1$ and $\Delta z_2$ are the out-of-plane displacements of the front and rear surfaces of the film, respectively; $\Delta t_s = \Delta z_2 - \Delta z_1$ is the geometrical thickness change of the sample; $\Delta\Lambda_0$ is the optical path length variation induced by the medium temperature, which is $\Delta\Lambda_0 = (z_R - z_g)\Delta n_m$ in our system; and $z_g$ is the depth of the glass window. A method has been mentioned in [10] for estimating $\Delta n_s$ and $\Delta t_s$ by using only $\Delta\Lambda_1$ and $\Delta\Lambda_3$. The method assumes that $\Delta\Lambda_2$ is negligible since it is much smaller than $\Delta\Lambda_1$ and $\Delta\Lambda_3$. However, two

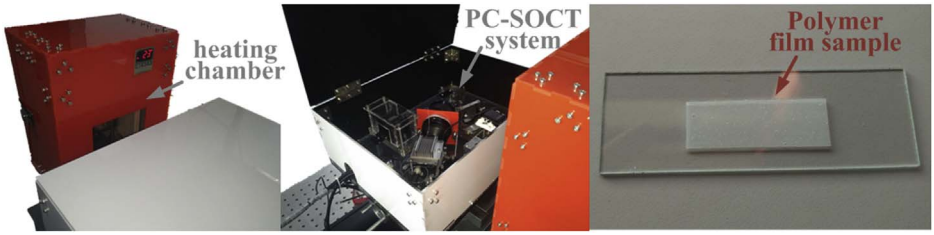


**Fig. 2.** Photographs of the PC-SOCT system, heating chamber, and film sample.