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Biaxial stress of precursor polymers using holographic interferometry

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Abstract

Holographic interferometry is applied to precursor films to determine their biaxial stresses. The theory of membrane deformation and vibrations is briefly reviewed to establish the relationship between stress and measured frequencies. Membranes for vibrational analysis are prepared using a circular precursor polymer. The biaxial stresses of the PHA, PAA, and 50/50 PHA/PAA are fairly constant with increasing frequencies: 6.85–7.61 MPa, 27.01–27.70 MPa, and 11.13–12.08 MPa, respectively. Regardless of the vibration modes, the biaxial stresses remain constant.

Keywords: Holographic interferometry; Precursor polymer; Biaxial stress

1. Introduction

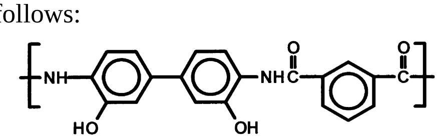
Since the concept of holographic interferometry was first proposed by Dennis Gabor in 1948 [1], holographic interferometry has been used in nodal analyses on vibrating objects [2–4]. Holographic interferometry was used extensively in various applications: the direct measurement of mechanical stresses [5–7], pressure stresses [8], vibrational excitation [9], shrinkage [10], swelling [11], and thermal stresses [12]. Holographic interferometry has also been used as a tool for stress measurement in polymer membranes [5,13,14]. To obtain the stress of the membrane using the bending strain, one must know the film thickness and the elastic constants of the membrane. The holographic interferometry method with vibrations presented here, however, measures the stress directly, and the only material parameter needed is the density of the film.

An introduction to the holographic interferometry technique for measurement of biaxial stresses in precursor membranes is presented in this article. Holographic interferometry is applied to measure the biaxial stresses of the precursor polymers and their polyblend.

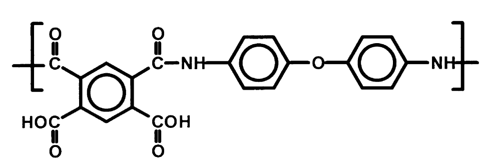
2. Experimental

2.1. Materials and blending

The polyhydroxyamide (PHA) was prepared by solution polymerization of 3,3′-dihydroxybenzidine and isophthaloyl dichloride [15]. Poly(amic acid) (PAA) was supplied by DuPont as a 15 wt.% solution in *N*,*N*-dimethylacetamide (DMAc). The chemical structures of PHA and PAA are as follows:



PHA



PAA

For a polyblend, PHA and PAA in solution were mixed at room temperature according to weight percentage. For 50/50 PHA/PAA in a blend, a solution in DMAc was prepared by mixing under vigorous agitation for one day. The concentration of the blended solution was 10% by weight. The polymer solution was coated onto a glass plate and dried in a vacuum oven at 60°C for one day. The blended film, while still on the glass plate, was then cleaned for five

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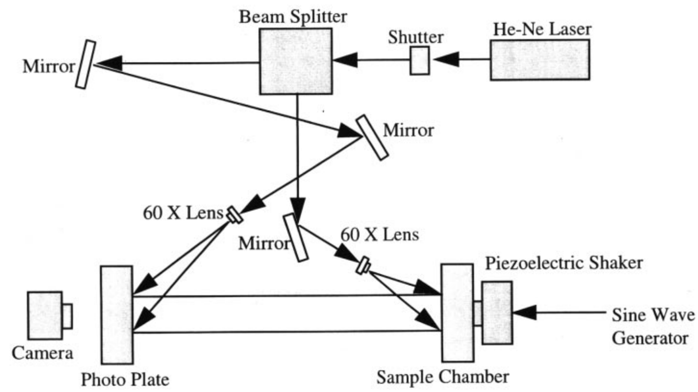

Fig. 1. Schematic diagram of the holographic interferometry equipment.

times in an ultrasonic cleaner for 30 min each. These films were removed with solvent, and dried again in a vacuum oven at 60°C for one day. Table 1 shows the general properties of PHA, PAA, and 50/50 PHA/PAA. As seen in Table 1, 50/50 PHA/PAA showed two endothermic peaks which indicate that the two precursors are not miscible with each other.

2.2. Sample preparation for biaxial stress measurement

A rigid circular steel washer was adhered to the flat precursor film using Super Glue and pressure was applied to ensure uniform adherence. After the glue was dried, the films on the washer were soaked in an ultrasonic cleaner with absolute ethyl alcohol. The film was dried in a vacuum oven at 60°C for a day in order to preserve the original state of stress.

2.3. Equipment of holographic interferometry

A membrane is placed in a fixture mounted rigidly in a Wilcoxon Research piezoelectric shaker, driven by a Wavetek Model 190 frequency generator connected to a Wilcoxon Research PA7 power amplifier. The frequency is monitored using a B and K Precision 80 MHz frequency counter. An image of the stationary membrane is recorded on a thermoplastic holographic plate using a Newport Research Corporation HC301 holographic camera. The light source used for this reflection hologram is a 5 mW helium–neon laser. A schematic of the holographic setup is shown in Fig. 1.

Table 1
General properties of the precursor polymers [18]

PHA/PAA (wt.%)	IV[a]	T^{b} (°C)	ΔH^{c} (J/g)	T_{D}^{d} (°C)
PHA (100/0)	1.32	319	221.1	577
PHA/PAA (50/50)	1.41	179, 318	54.7, 66.0	537
PAA (0/100)	2.01	178	122.1	533

[a] Inherent viscosities of the PHA and 50/50 PHA/PAA were measured at 30°C by using 0.2 g/dL solution in NMP. Intrinsic viscosity of the PAA was measured at 30°C by using 15 wt.% solution in DMAc.
[b] Minimum point in endothermic curve.
[c] Endothermic enthalpy change.
[d] Initial decomposition temperature.

3. Results and discussion

Holographic interferometry is based on classical vibration theory [5,16]. The free vibration of a membrane in vacuum is described by the differential equation,

$$\sigma\nabla^2 u = \rho(\partial^2 u/\partial t^2), \tag{1}$$

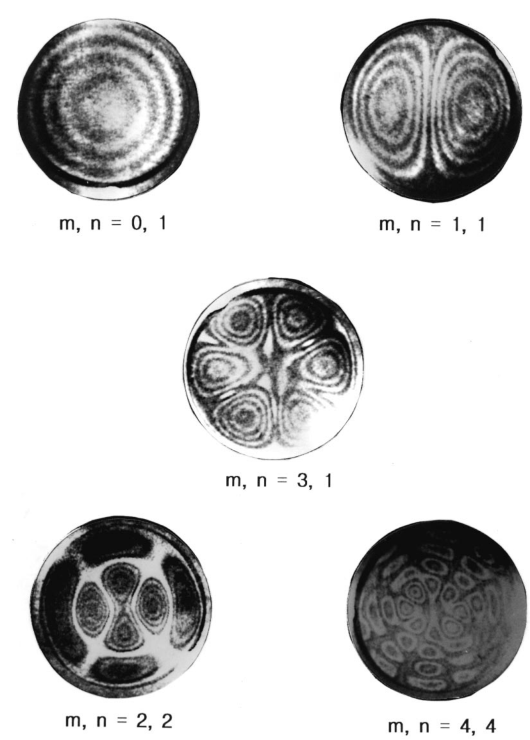

Fig. 2. Mode shapes of a PHA membrane observed using holographic interferometry.

Table 2
Residual biaxial stress for the precursor membranes tested in a vacuum

No	*m, n*	Z_{mn}	PHA		50/50 PHA/PAA		PAA	
			Freq. (sec^{-1})	Stress (MPa)	Freq. (sec^{-1})	Stress (MPa)	Freq. (sec^{-1})	Stress (MPa)
1	0, 1	2.405	1385	7.31	1683	11.13	2650	27.70
2	1, 1	3.831	2102	6.85	2719	11.44	4185	27.22
3	2, 1	5.138	2846	6.97	3746	12.08	5635	27.44
4	3, 1	6.380	3612	7.28	4592	11.77	7013	27.56
5	4, 1	7.586	4390	7.61	5510	11.99	8315	27.41
6	2, 2	8.417					9170	27.01

where, σ is the biaxial stress (in N/m^2), u the out of plane displacement, ρ the density (in kg/m^3), ∇^2 the LaPlacian operator, $\partial^2 u/\partial t^2$ the second time derivative of the out of plane displacement.

If the membrane is attached at its circumference to a rigid support, and the boundary condition at $r = R$ (where R is the outer radius of the membrane) is $u = 0$, then the solution to Eq. (1) for a circular membrane is

$$\sigma = \rho(\omega_{mn} R/Z_{mn})^2, \quad (2)$$

where, σ is the biaxial stress (in N/m^2), ρ the density (in kg/m^3), ω_{mn} the angular frequency (in rad/s), R the radius of membrane (in m) Z_{mn} the nth zero of the mth order Bessel function.

In Eq. (2), the biaxial stress in the membrane is directly related to the frequencies at which the membrane resonates. The zeroes of the integer order Bessel functions were tabulated in literature [5,17]. Therefore, if the state of stress in the membrane is preserved, only the measure of material density is required to directly determine the biaxial stress. As mentioned earlier, this is advantageous because no constitutive assumptions are necessary and knowledge of the elastic constants of the material is not required.

A summary of the stress values, their corresponding mode number, and their resonant frequency of vibration is given in Table 2. The mode number is simply an integer that indicates the sequence of the observed modes of vibration of the membrane. Generally, for a given sample, the many modes of vibration can be seen with each mode occurring at a unique resonance frequency. However, all of them give the same value of stress when calculated from Eq. (2).

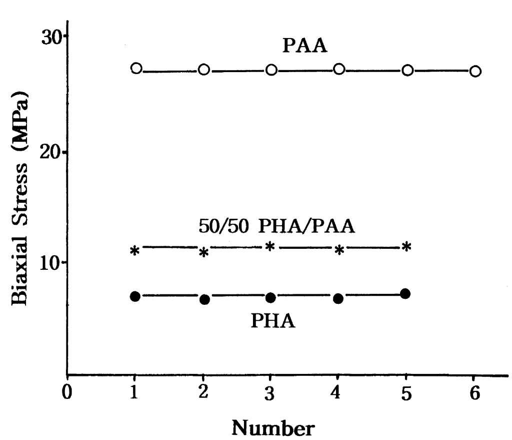

Fig. 3. Calculated biaxial stresses for the PHA, PAA and 50/50 PHA/PAA membranes as a function of the number shown in Table 2.

The radius of the membrane is 2.050 cm and the film density is 1.368 g/cm^3 for PHA, 1.375 g/cm^3 for PAA, and 1.370 g/cm^3 for 50/50 PHA/PAA, respectively. The number of the zero and the order of the Bessel function is determined directly from the observed vibration pattern at a given frequency. The indices m and n represent the number of radial and tangential nodal lines, respectively. The relationship between frequency and stress is independent of film thickness and all material parameters except density.

As seen in Table 2, the biaxial stresses of the PHA, PAA, and 50/50 PHA/PAA are 6.85–7.61 MPa, 27.01–27.70 MPa, and 11.13–12.08 MPa, respectively, with increasing frequencies. In particular, the biaxial stress of the 50/50 PHA/PAA was lower than the value expected from an additivity rule. This can be attributed to poor miscibility of the two precursor polymers in blending [18]. For these samples, we were able to observe vibration patterns from some of the modes of vibration in vacuum. A few examples of typical holographic patterns are shown in Fig. 2. For the same membrane sample, higher resonant modes can be excited at specific resonant frequencies, but all of them show constant stress value when substituted in Eq. (2). A plot of biaxial stress for the PHA, PAA, and the blended membrane is shown in Fig. 3.

In conclusion, holographic interferometry is a valuable technique for resolving the state of stress in membranes. Special sample preparation techniques were designed to preserve the state of stress in the membrane. Measured and predicted mode shapes are in very close agreement.

Acknowledgements

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References

[1] Gabor D. Nature 1948;161(4098):777.
[2] Aprahamian R, Evensen DA, Mixson JS, Jacoby JL. Experimental Mechanics 1971;357.
[3] Heflinger LO, Wuerker RF, Brooks RE. J Appl Phys 1966;37(2):642.
[4] Ito A, Kashiwagi T. Appl Opt 1987;26(14):954.
[5] Maden MA, PhD Thesis, University of Massachusetts at Amherst, MA, 1992.
[6] Maden MA, Jagota A, Mazur S, Farris RJ. J Am Ceram Soc 1994;77:625.
[7] Tong QA, Maden MA, Jagota A, Farris RJ. J Am Ceram Soc 1994;77:636.
[8] Kussmaul K, Ettemeyer A. Nuclear Engineering and Design 1987;102:307.
[9] Fallstrom KE, Gustavsson H, Mplin NE, Wahlin A. Experimental Mechanics 1989;378.
[10] Jung P. SPIE, Optic in Engineering Measurement 1985;599:32.
[11] Goldberg JE, O'Toole KM, Roper H. J Test and Eval 1975;3(4):263.
[12] Gilbert JA, Vedder DT. Experimental Mechanics 1981;138.
[13] Chang J-H, Chen MJ, Farris RJ. Polymer 1998;23:5649.
[14] Chen MJ. PhD Thesis, University of Massachusetts at Amherst, MA, 1997.
[15] Kubota T, Nakanishi R. J Polym Sci B 1964;2:655.
[16] Vrtis J, Farris RJ. Mat Res Soc Symp Proc 1994;338:527.
[17] Arfken G. Mathematical Methods for Physicists. 3. New York: Academic Press, 1985 pp. 572–575.
[18] Chang J-H, Farris RJ, Polym Eng and Sci (in press).