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Using DMA to simultaneously acquire Young's relaxation modulus and time-dependent Poisson's ratio of a viscoelastic material

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Abstract

A method to obtain the Young's relaxation modulus and time-dependent Poisson's ratio simultaneously by using DMA is developed with the assumption of constant bulk modulus instead of constant Poisson's ratio. The constant bulk modulus is then calculated by either instantaneous response or the equilibrium response of the time-dependent Poisson's ratio. The modulating Young's moduli and characteristic times that measured by DMA are corrected analytically by using the developed formulas. The time-dependent Poisson's ratio is then obtained from the corrected modulating Young's moduli and the constant bulk modulus. As an application example, the method is applied to the DMA measurement of an epoxy molding compound (EMC). Although the correction to Young's relaxation modulus is very small, the viscoelastic Poisson's ratio varies significantly over time from 0.4 to 0.496, and can’t be assumed as a constant.

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Keywords: dynamic mechanical analysis; Young's relaxation modulus; time-dependent Poisson's ratio; relaxation-creep duality representation; Prony series

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1. Introduction

Dynamic mechanical analyzer (DMA) is a powerful instrument that is widely used in the electronic package to measure the viscoelasticity or shrinkage behaviors of molding compound or underfill [1-7]. Typically, a DMA instrument has various modes of operation, including shear mode, bending mode (dual/single cantilever, 3-point bending), compression mode, and tension mode. The first mode is used to measure the viscoelastic shear modulus, and the others are used to measure the viscoelastic Young's modulus [8].

For bending modes, the Young's complex modulus is calculated by using [8]

$$E^*_{\mathrm{DMA}}(\omega) = K_S(\omega) f_c \frac{L^3}{I}\left[1+\frac{12}{5}(1+\nu)\left(\frac{h}{L}\right)^2\right], \tag{1}$$

where $K_s(\omega)$ is the measured stiffness, f_c is a constant that depends on the bending mode, h is the sample thickness, L is the sample half length, I is the sample moment of inertia, and ν is the Poisson's ratio. It is noted that the Young's complex modulus is not only depending on the geometric parameters, but also on the Poisson's ratio. The Poisson's ratio was frequently assumed to be a constant in the literatures [9-14]. However, the constant Poisson's ratio implies the ratio of the bulk relaxation modulus, $K(t)$, and the shear relaxation modulus, $G(t)$, is a constant; that is, $K(t) = \frac{2(1+\nu)}{3(1-2\nu)}G(t)$ [15] . This relation also implies that both bulk and shear relaxation moduli share the same relaxation times. However, experimental data showed that for most materials the relaxation times of bulk relaxation moduli are much higher than those of shear relaxation moduli [16-18]. Therefore, the assumption of constant Poisson's ratio is inappropriate. Instead, the constant bulk modulus is a more applicable assumption [15,20-22].

In this work, correction to Eq. (1) is derived to incorporate the effect of time-dependent Poisson's ratio. From this correction, the Young's relaxation modulus and time-dependent Poisson's ratio can be obtained simultaneously from a single DMA test. Section 1 gives the description of the time-dependent effect of Poisson's ratio and the assumption adopted in DMA measurement. In Section 2, the correction of Young's complex modulus is first derived, and the time-dependent Poisson's ratio is then calculated from the Young's relaxation modulus. The implementation of this correction is carried out in Section 3 to DMA measurements of a molding compound. The conclusion is given in Section 4.

2. The corrections to Young's relaxation modulus and time-dependent Poisson's ratio

Two parameters in viscoelastic model are frequently used to represent the elastic response: one is the instantaneous parameter, and the other is the permanent parameter. The instantaneous parameter is usually viewed as the instantaneous elastic response of a viscoelastic fluid, while the permanent parameter as the equilibrium elastic response of a viscoelastic solid. The instantaneous parameters of Young's relaxation modulus and time-dependent Poisson's ratio are denoted by

$$E_0 = E(0), \quad \nu_0 = \nu(0); \tag{2}$$

while the permanent parameters of Young's relaxation modulus and time-dependent Poisson's ratio are given by

$$E_\infty = E(\infty), \quad \nu_\infty = \nu(\infty). \tag{3}$$

2.1. Assigning ν_0 as the elastic Poisson's ratio

Instead of assuming the constant Poisson's ratio in Eq. (1), we use the constant bulk modulus to reformulate the Eq. (1) as

$$E^*(\omega) = K_S(\omega) f_c \frac{L^3}{I}\left[1+\frac{12}{5}\left(\frac{3}{2}-\frac{E^*(\omega)}{6K}\right)\left(\frac{h}{L}\right)^2\right], \tag{4}$$

where K is calculated from the instantaneous parameters and is given by

$$K = \frac{E_0}{3(1-2\nu_0)}, \tag{5}$$

and

$$E_0 = E(0) = E_{\mathrm{DMA}}(0) \cdot \tag{6}$$

In Eqs. (4) and (5), the initial value of the corrected Young's relaxation modulus coincides to the uncorrected one. The ν appears in Eq. (1) is realized as the instantaneous parameter of time-dependent Poisson's ratio, i.e., ν_0. By dividing Eq. (1) by Eq. (4), and rearranging the result, it follows:

$$E^*(\omega) = \frac{\left[1+\frac{12}{5}\left(\frac{h}{L}\right)^2\frac{3}{2}\right]E^*_{\mathrm{DMA}}(\omega)}{1+\frac{12}{5}\left(\frac{h}{L}\right)^2\left[\frac{3}{2}+\frac{1}{6K}\left(E^*_{\mathrm{DMA}}(\omega)-E_0\right)\right]}. \tag{7}$$

If $E_{\mathrm{DMA}}(t)$ can be expressed as the 2-element Prony series (the extension to N-element Prony series is straightforward), then its representation in Laplace transform domain (s-domain) is given by [20]

$$s\widetilde{E}_{\mathrm{DMA}}(s) = \frac{E_0^{\mathrm{DMA}}s^2 + E_1^{\mathrm{DMA}}\left(\frac{1}{\tau_1^{\mathrm{DMA}}}+\frac{1}{\tau_2^{\mathrm{DMA}}}\right)s + E_\infty^{\mathrm{DMA}}\frac{1}{\tau_1^{\mathrm{DMA}}\tau_2^{\mathrm{DMA}}}}{s^2+\left(\frac{1}{\tau_1^{\mathrm{DMA}}}+\frac{1}{\tau_2^{\mathrm{DMA}}}\right)s+\frac{1}{\tau_1^{\mathrm{DMA}}\tau_2^{\mathrm{DMA}}}}. \tag{8}$$

Replacing $E^*(\omega)$ by $s\widetilde{E}(s)$ and $E^*_{\mathrm{DMA}}(\omega)$ by $s\widetilde{E}_{\mathrm{DMA}}(s)$, the corrected $s\widetilde{E}(s)$ is obtained via Eq. (8):

$$s\widetilde{E}(s) = \frac{E_0 s^2 + E_1\left(\frac{1}{\tau_1}+\frac{1}{\tau_2}\right)s + E_\infty\frac{1}{\tau_1\tau_2}}{s^2+\left(\frac{1}{\tau_1}+\frac{1}{\tau_2}\right)s+\frac{1}{\tau_1\tau_2}}, \tag{9}$$

where

$$E_0 = E_0^{\mathrm{DMA}}, \quad E_1 = \frac{\left[1+\frac{12}{5}\left(\frac{h}{L}\right)^2\frac{3}{2}\right]E_1^{\mathrm{DMA}}}{1+\frac{12}{5}\left(\frac{h}{L}\right)^2\left[\frac{3}{2}+\frac{E_1^{\mathrm{DMA}}-E_0}{6K}\right]}, \quad E_\infty = \frac{\left[1+\frac{12}{5}\left(\frac{h}{L}\right)^2\frac{3}{2}\right]E_\infty^{\mathrm{DMA}}}{1+\frac{12}{5}\left(\frac{h}{L}\right)^2\left[\frac{3}{2}+\frac{E_\infty^{\mathrm{DMA}}-E_0}{6K}\right]}, \tag{10}$$

and

$$\frac{1}{\tau_1}+\frac{1}{\tau_2} = \frac{1+\frac{12}{5}\left(\frac{h}{L}\right)^2\left[\frac{3}{2}+\frac{E_1^{\mathrm{DMA}}-E_0}{6K}\right]}{1+\frac{12}{5}\left(\frac{h}{L}\right)^2\frac{3}{2}}\left(\frac{1}{\tau_1^{\mathrm{DMA}}}+\frac{1}{\tau_2^{\mathrm{DMA}}}\right), \quad \frac{1}{\tau_1\tau_2} = \frac{1+\frac{12}{5}\left(\frac{h}{L}\right)^2\left[\frac{3}{2}+\frac{E_\infty^{\mathrm{DMA}}-E_0}{6K}\right]}{1+\frac{12}{5}\left(\frac{h}{L}\right)^2\frac{3}{2}}\frac{1}{\tau_1^{\mathrm{DMA}}\tau_2^{\mathrm{DMA}}}. \tag{11}$$

In addition, the relaxation-creep duality representation of $E(t)$ can be expressed as

$$E(t) = a_1^E\left[E_0\,\mathrm{e}^{-t/\tau_1} + E_\infty\,\bar{\mathrm{e}}^{-t/\tau_1}\right] + a_2^E\left[E_0\,\mathrm{e}^{-t/\tau_2} + E_\infty\,\bar{\mathrm{e}}^{-t/\tau_2}\right], \tag{12}$$

where

$$a_1^E = \frac{X_1^E/\tau_1 - X_2^E/\tau_2}{1/\tau_1 - 1/\tau_2}, \quad a_2^E = \frac{X_2^E/\tau_1 - X_1^E/\tau_2}{1/\tau_1 - 1/\tau_2}, \quad X_1^E = \frac{E_0-E_1}{E_0-E_\infty}, \quad X_2^E = \frac{E_1-E_\infty}{E_0-E_\infty}, \quad \bar{\mathrm{e}}^{-t/\tau} = 1-\mathrm{e}^{-t/\tau}. \tag{13}$$

The modification of elastic Poisson's ratio to be time-dependent can be implemented by calculating the modulating constants, given by [20]

$$\nu_0 = \frac{1}{2} - \frac{E_0}{6K}, \quad \nu_1 = \frac{1}{2} - \frac{E_1}{6K}, \quad \nu_\infty = \frac{1}{2} - \frac{E_\infty}{6K}. \tag{14}$$

The characteristic times of $\nu(t)$ are the same as those calculated from Eqs. (11). Similar to $E(t)$, the relaxation-creep duality representation of $\nu(t)$ can be expressed as

$$\nu(t) = a_1^\nu \left[\nu_0 \, e^{-t/\tau_1} + \nu_\infty \, \bar{e}^{-t/\tau_1}\right] + a_2^\nu \left[\nu_0 \, e^{-t/\tau_2} + \nu_\infty \, \bar{e}^{-t/\tau_2}\right], \tag{15}$$

where

$$a_1^\nu = \frac{X_1^\nu/\tau_1 - X_2^\nu/\tau_2}{1/\tau_1 - 1/\tau_2}, \quad a_2^\nu = \frac{X_2^\nu/\tau_1 - X_1^\nu/\tau_2}{1/\tau_1 - 1/\tau_2}, \quad X_1^\nu = \frac{\nu_0 - \nu_1}{\nu_0 - \nu_\infty}, \quad X_2^\nu = \frac{\nu_1 - \nu_\infty}{\nu_0 - \nu_\infty}. \tag{16}$$

2.2. Assigning ν_∞ as the elastic Poisson's ratio

When the elastic Poisson's ratio is regarded as ν_∞, Eq. (4) is still valid, but K is calculated from the permanent parameters:

$$K = \frac{E_\infty}{3(1 - 2\nu_\infty)}, \tag{17}$$

where

$$E_\infty = E(\infty) = E_{\mathrm{DMA}}(\infty). \tag{18}$$

In this case, the equilibrium value of the corrected Young's relaxation modulus coincides to the uncorrected one, and in Eq. (1), ν is realized as the permanent parameter of the time-dependent Poisson's ratio.

By following a similar derivation as in Section 2.1, the corrected Young's complex modulus can be expressed as

$$E^*(\omega) = \frac{\left[1 + \frac{12}{5}\left(\frac{h}{L}\right)^2 \frac{3}{2}\right] E_{\mathrm{DMA}}^*(\omega)}{1 + \frac{12}{5}\left(\frac{h}{L}\right)^2 \left[\frac{3}{2} + \frac{1}{6K}\left(E_{\mathrm{DMA}}^*(\omega) - E_\infty\right)\right]}. \tag{19}$$

If $E_{\mathrm{DMA}}(t)$ can be expressed as the 2-element Prony series, Eqs. (8), (9), (12)-(16) are still applicable, but the corrections of parameters are changed to

$$E_0 = \frac{\left[1 + \frac{12}{5}\left(\frac{h}{L}\right)^2 \frac{3}{2}\right] E_0^{\mathrm{DMA}}}{1 + \frac{12}{5}\left(\frac{h}{L}\right)^2 \left[\frac{3}{2} + \frac{E_0^{\mathrm{DMA}} - E_\infty}{6K}\right]}, \quad E_1 = \frac{\left[1 + \frac{12}{5}\left(\frac{h}{L}\right)^2 \frac{3}{2}\right] E_1^{\mathrm{DMA}}}{1 + \frac{12}{5}\left(\frac{h}{L}\right)^2 \left[\frac{3}{2} + \frac{E_1^{\mathrm{DMA}} - E_\infty}{6K}\right]}, \quad E_\infty = E_\infty^{\mathrm{DMA}}, \tag{20}$$

and

$$\frac{1}{\tau_1} + \frac{1}{\tau_2} = \frac{1 + \frac{12}{5}\left(\frac{h}{L}\right)^2 \left[\frac{3}{2} + \frac{E_1^{\mathrm{DMA}} - E_\infty}{6K}\right]}{1 + \frac{12}{5}\left(\frac{h}{L}\right)^2 \left[\frac{3}{2} + \frac{E_0^{\mathrm{DMA}} - E_\infty}{6K}\right]} \left(\frac{1}{\tau_1^{\mathrm{DMA}}} + \frac{1}{\tau_2^{\mathrm{DMA}}}\right), \quad \frac{1}{\tau_1 \tau_2} = \frac{1 + \frac{12}{5}\left(\frac{h}{L}\right)^2 \frac{3}{2}}{1 + \frac{12}{5}\left(\frac{h}{L}\right)^2 \left[\frac{3}{2} + \frac{E_0^{\mathrm{DMA}} - E_\infty}{6K}\right]} \frac{1}{\tau_1^{\mathrm{DMA}} \tau_2^{\mathrm{DMA}}}. \tag{21}$$

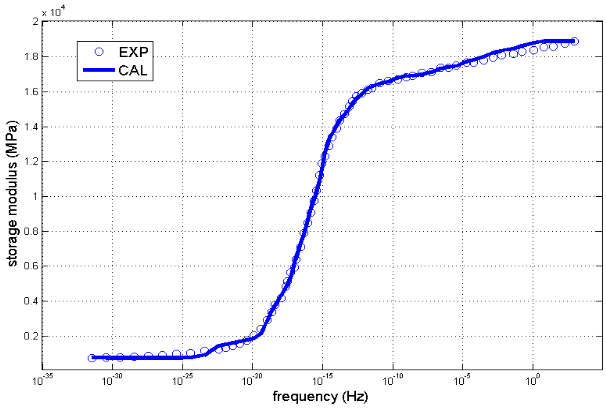

Fig. 1. Master curve for EMC, reference temperature is 30 °C

3. Viscoelastic characterization of epoxy molding compound

As an application example, the correction method described in Section 2 is applied to the DMA measurement of an EMC. The DMA experiment is conducted under a 3-point bending configuration. Dimension of the EMC specimen is 60 mm×10 mm×2 mm. The distance between the outer clamps is 35 mm and the bending oscillation amplitude is 10 μm. The sampling points of frequencies are 1, 5, 10, 20, 50, 100, 150 and 200 Hz, while the temperature range is from -30 to 300 °C with constant heating rate of 3 °C/min. The time-temperature superposition (TTS) principle is applied to construct the storage modulus master curve with a reference temperature of 30 °C as shown in Fig. 1. The corresponding shift factors is modeled by using the Williams-Landel-Ferry (WLF) model, with the fitted model constants C_1 = 437.63 and C_2 = 2558.87. The fitted relaxation-creep duality representation parameters (superscripted by DMA) are shown in Tables 1 and 2.

The only material property that is inputted into the DMA program for viscoelastic property calculations is the elastic Poisson's ratio, which is assigned with the value $\nu = 0.4$. Since the EMC behaves more like rubber after loaded for an extended time, the value of 0.4 is not adequate for ν_∞, and should be considered as ν_0. The corresponding K can be calculated as 31.5 GPa, and the corrected modulating Young's moduli and characteristic times can be obtained according to Eqs. (10) and (11) (extended to 25 elements) and are shown in Tables 1 and 2. It can be seen from Tables 1 and 2 that the corrections are insignificant, especially for characteristic times. However, when Eq. (14) (extended to 25 elements) is used to correct the Poisson's ratio, it is shown that, as time increases, the $\nu(t)$ changes from 0.4 to 0.496. This trend is proper for the EMC which has a large-time behavior like rubber.

Table 1. The measured values of modulating Young's moduli (superscripted by DMA) and the corrected values of modulating Young's moduli and modulating Poisson's ratios

n	0	1	2	3	4	5	6	7	8
E_n^{DMA} (MPa)	18900	18779	18538	18398	18089	17857	17571	17435	17145
E_n (MPa)	18900	18784	18543	18403	18093	17861	17575	17438	17147
ν_n	0.400	0.401	0.402	0.403	0.404	0.405	0.407	0.408	0.409
n	9	10	11	12	13	14	15	16	17
E_n^{DMA} (MPa)	17035	16904	16632	16382	15752	14691	13374	10165	7502
E_n (MPa)	17037	16905	16633	16383	15752	14692	13374	10165	7502
ν_n	0.410	0.411	0.412	0.413	0.417	0.422	0.429	0.446	0.460
n	18	19	20	21	22	23	24	∞	
E_n^{DMA} (MPa)	4980	3627	2052	1776	1582	1410	878	750	
E_n (MPa)	4980	3627	2052	1776	1582	1410	878	751	
ν_n	0.474	0.481	0.489	0.491	0.492	0.493	0.495	0.496	

Table 2. The measured (superscripted by DMA) and corrected values of characteristic times

n	1	2	3	4	5	6	7	8	9
τ_n^{DMA} (s)	1.000E+00	1.000E+01	1.000E+02	1.000E+03	1.000E+04	1.000E+05	1.000E+06	1.000E+07	1.000E+08
$\tau_n - \tau_n^{DMA}$ (s)	1.483E-06	3.805E-05	1.433E-04	4.834E-03	3.002E-02	4.409E-01	1.328E+00	4.728E+01	1.143E+02
n	10	11	12	13	14	15	16	17	18
τ_n^{DMA} (s)	1.000E+09	1.000E+10	1.000E+11	1.000E+12	1.000E+13	1.000E+14	1.000E+15	1.000E+16	1.000E+17
$\tau_n - \tau_n^{DMA}$ (s)	1.621E+03	4.144E+04	2.736E+05	8.766E+06	1.527E+08	1.530E+09	4.986E+10	3.658E+11	3.746E+12
n	19	20	21	22	23	24	25		
τ_n^{DMA} (s)	1.000E+18	1.000E+19	1.000E+20	1.000E+21	1.000E+22	1.000E+23	1.000E+24		
$\tau_n - \tau_n^{DMA}$ (s)	1.629E+13	2.513E+14	1.488E+14	2.657E+15	1.674E+16	8.874E+17	1.090E+18		

4. Conclusions

A method is developed for obtaining the Young's relaxation modulus and time-dependent Poisson's ratio simultaneously by using DMA with the assumption of constant bulk modulus instead of constant Poisson's ratio. This method is based on the relaxation-creep duality representation, which is a variant of Prony series. The original elastic Poisson's ratio can be viewed as the instantaneous or the equilibrium response of the time-dependent Poisson's ratio. The constant bulk modulus is then calculated by either instantaneous or the equilibrium response of the time-dependent Poisson's ratio. The modulating Young's moduli and characteristic times from DMA measurement are corrected analytically by using the developed formulas. In addition, the time-dependent Poisson's ratio is obtained from the corrected modulating Young's moduli and the constant bulk modulus.

The viscoelastic property of the EMC used for electronic packaging is considered as an example. For the DMA program, a value of 0.4 is inputted as the elastic Poisson's ratio. From the DMA measurement, the uncorrected Young's relaxation modulus is obtained and expressed by using a 25-element relaxation-creep duality representation. Corrections are made to both modulating moduli and characteristic times by using the developed method. Furthermore, the modulating constants for the time-dependent Poisson's ratio are calculated. Although the correction to Young's relaxation modulus is very small, the time-dependent Poisson's ratio varies largely from 0.4 to 0.496, and can't be assumed as a constant.

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