Sound Absorption in the Low Audible Frequency Range of Microfibrous Parylene-C Thin Films

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Microfibrous thin films (μFTFs) of Parylene C are deposited to a thicknesses of about 100 μm by physicochemical vapor deposition with the intention of determining the sound absorption of these films in the lower audible frequency range. The objective is to determine the sound absorption by the μFTFs by using dynamic loading experiments. The μFTFs were subjected to cyclic elastic loads in the frequency range of 5 to 200 Hz over a temperature range of 25 to 50 °C to determine their dynamic moduli and thus extract the Parylene-C μFTFs sound absorption properties. The absorption coefficient of microfibrous Parylene-C is found to be weakly dependent on temperature, however it increases with increasing frequency. Peaks in the spectra of the absorption coefficient were attributed to resonant coupling between incident sound waves and vibrating microfibers.

1. INTRODUCTION

Microfibrous thin films (μFTFs) are important materials in optical, chemical, and biochemical applications.[1] μFTFs are fabricated by either physical or chemical vapor deposition methods with oblique-angle deposition techniques.[1,2] Different material types, including metals, ceramics, and polymers were successfully sculptured by using these techniques.[1] The typical structural μFTF morphologies produced are shown in the scanning electron microscope (SEM) images in Fig. 1.

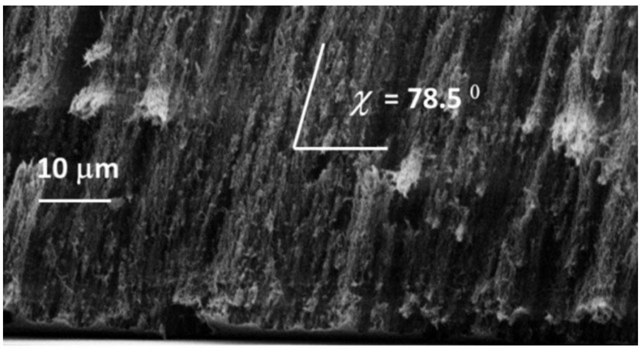

Figure 1. A typical FESEM micrograph of a Parylene-C μFTF.

Parylene-C, a polymer material, has often been used as moisture-impermeable coating in medical devices and electronics.[3] For these medical and electronic applications, Parylene-C was prepared in bulk form, i.e., as a dense homogeneous film, by using chemical vapor deposition.[4] However, the μFTF-growth of Parylene-C introduced periodicity. Therefore, it aided in the the investigation of acoustic and electromagnetic wave propagation characteristics and availed the possibility of acoustics optical applications. These applications required the investigation of the mechanical and dielectric properties of Parylene-C μFTFs.

In this article, μFTFs of Parylene-C were fabricated using a physicochemical vapor deposition process and examined using dynamic mechanical loading, acoustic insertion loss, and transmission spectroscopy.[5,6] The storage, loss, moduli, and absorption coefficient for the Parylene-C μFTFs were obtained as a function of temperature and frequency. There are several studies on the effects of nano/microstructures on sound absorption. However, this is the first such study on microfibrous thin films.[7–9]

2. THE EXPERIMENTAL PROCEDURE

The microfibers of Parylene C used in this work were fabricated using a physicochemical vapor deposition process([5,6]) and used a custom made PDS2010 Labcoater. Four grams of a Parylene-C dimer were first vaporized at 175 °C and then pyrolyzed at 690 °C into a monomer vapor. A collimated flux of the monomer vapor was directed from a nozzle at 45 ° towards a planar 2 cm × 2 cm Si substrate in a low-pressure chamber maintained at 175 °C and 28 mTorr. Finally, the thin film was removed from the silicon substrate using a razor.

After being removed, the morphologies of the grown samples were first examined using a field-emission scanning electron microcopy (FESEM) with a Model LEO 1530, Carl Zeiss microscope. After the morphology examination, each sample was held between the two appropriately spaced grips of a tension clamp and subjected to cyclic loading in a dynamic mechanical analyzer (DMA). The DMA used was the Model Q800 equipment, which was made by TA Instruments and used a "Multi-Frequency Strain" module. The tension-clamp was calibrated with a thin steel sheet of known compliance and dimensions. The measured experimental temperature range was between 25 °C and 150 °C in steps of 5 °C. At each temperature, a cyclic strain of amplitude 0.046 % (elastic regime) was set for frequencies between 5 and 200 Hz in increments of 5 Hz.

3. RESULTS AND DISCUSSION

From FESEM, the thicknesses of the samples were found to be in the range of 100 to 110 microns and the microfiber diameters were determined to be about 5 μm inclined at about 80 °

to the substrate plane. Fig. 1 shows a typical SEM micrograph observed in the film. To vary the inclination of the microfiber, one needs to change the angle of incidence of the collimated flux on the substrate.

In this work, we attempted to determine the acoustic absorption coefficient α of the Parylene-C μFTF in order to explore its application in acoustic applications, as discussed in the following subsection. The absorption coefficient α obtained in this study determined sound absorption through the relation $I_x = (I_o - I_R)e^{-\alpha x}$, where I_o was the sound intensity incident on the film's surface, I_R was the reflected sound intensity at $x = 0$, and $I(x)$ was the intensity at distance x below the surface. However, determining α for films as thin as the ones grown in this work was challenging and the method employed here used dynamic loading. For a linear material subjected to cyclic loading at frequency f a phase shift δ existed between stress σ and strain ϵ, which are given by:

$$\sigma = \sigma_0 \sin(\omega t) \text{and} \in = \epsilon_0 \sin(\omega t + \delta); \quad (1)$$

where t is time, σ_o and ϵ_o were the respective amplitudes of stress and strain, $\omega = 2\pi f$ was the angular frequency, and δ was a phase shift. The two elastodynamic moduli were the storage (elastic) modulus $E' = \frac{\sigma_o}{\epsilon_o}\cos\delta$ and the loss modulus $E" = \frac{\sigma_o}{\epsilon_o}\sin\delta$.[10] The absorption coefficient, α, was obtained from the phase shift δ using the relation:

$$\alpha = \frac{\omega}{2c}\tan\sigma \text{where} c = \sqrt{\frac{E'}{\rho}}; \quad (2)$$

where ρ was the film density.

We noted that α obtained in these experiments was extracted from an elastic and loss moduli that were determined by the stress-strain parameters on the plane of the substrate at an average angle of about 80 ° to the microfibers. This was expected to be different from α perpendicular to the substrate, i. e., making about 10 ° to the microfiber axis. The reason for this was the anisotropic nature of the film morphology, as is apparent from the SEM in Fig. 1. Also, α was expected to vary with χ, since the microfiber inclination to the substrate and the spacing between the microfibers were expected to vary with χ. Currently experiments are in progress in our laboratory to study changes in α with χ.

In an earlier study of Parylene-C μFTFs of similar thicknesses, we found that the glass transition temperature was $T_g = 65$ °C.[11] This was in contrast to the T_g reported between 30 °C and 60 °C in films of thicknesses about 20 μm and 92 °C for about 5 μm-thick films of dense (non-fibrous) Parylene-C.[12,13] This leads to the suggestion that the surface area of microfibrous thin film may be the reason for the differences of T_g between dense and microfibrous thin films. Therefore, the acoustic absorption coefficient data shown in Fig. 2 are limited to temperatures below T_g. In Fig. 2 we observed that the dependence of α on temperature was somewhat weak. However, there was a much stronger dependence of α on frequency in the range 5 Hz to 200 Hz measured in these experiments. α increased with increasing f and this increase was an order of magnitude as the frequency increased from 5 Hz to 200 Hz.

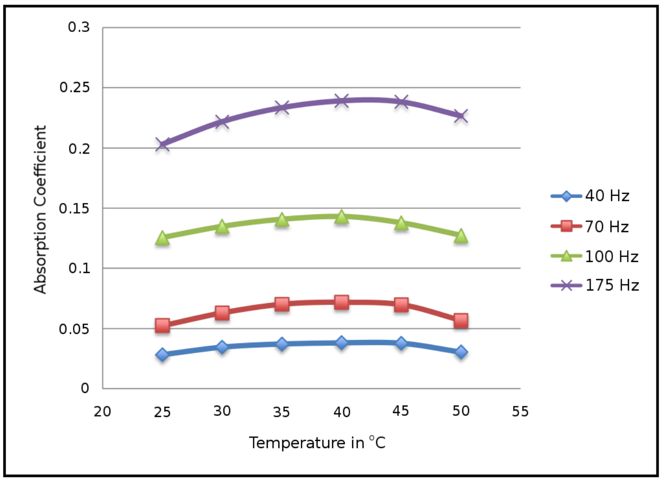

Figure 2. The acoustic absorption coefficient α in about 100 μm-thick Parylene-C μFTFas a function of temperature and frequency f in the low audible frequency range. α is in m^{-1}.

Figure 3 offers a closer look at the dependence of the absorption coefficient on f. Figure 3 features three distinct peaks in the 75–200 Hz frequency range at temperatures between 25 °C and 50 °C. The strongest middle peak occurs at f about 125 Hz with two smaller peaks: one below 100 Hz and the other above 150 Hz.

For a square μFTF film the stiffness constant, k, may be determined using the method described by Lintymer et al.[14]

$$k = \frac{Ed^4 \cos\beta}{n(d' + b)\Lambda^3 \tan^2\beta}; \quad (3)$$

where E is Young's modulus of the film, β was the angle the column made with the horizontal plane, d was the column diameter, d' was the longer axis of the elliptical cross section projection in the horizontal plane, b was the distance between two adjacent columns, Λ was the single period thickness, and n was the number of periods. By using values estimated from the film SEM micrographs, one gets $k \approx 8$ Nm^{-1}. The density of Parylene-C of 1289 kg/m^3, when used with the geometry of the μFTF sample, held between the two grips and yielded a film mass of $m = 1.36\times10^{-5}$ kg. The resonant frequency f_o for a microfiber of stiffness k and carrying a load m was given by:

$$\omega_0 = 2\pi f_0 = \sqrt{\frac{k}{m}} = 244 \text{ Hzor} f_0 = 122 \text{ Hz}. \quad (4)$$

This value for f_o agreed very well with the position of the largest peak in the frequency f at about 125 Hz. Therefore, the large peak in the absorption coefficient at f, which was about 125 Hz, was attributed to the resonant coupling between the incident sound waves and the vibrating microfibers. The remaining two peaks were suggested to arise from two additional secondary resonance frequencies, presumably from variations in the μFTF morphology.

4. CONCLUSIONS

We used vapor deposition to grow about 100 μm of microfibrous thin films (μFTFs) of Parylene C on top of a Si substrate.

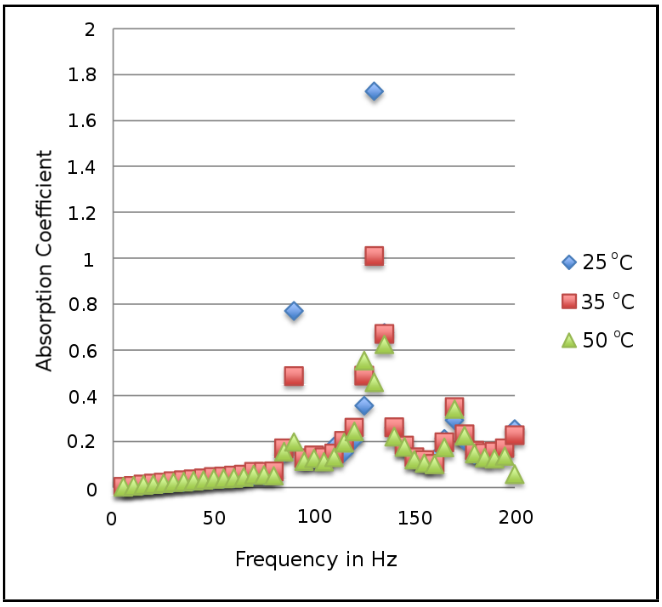

Figure 3. The acoustic absorption coefficient α in about 100 µm-thick Parylene-CµFTFas a function of frequency f and temperature. α is in m^{-1}.

The film microfibers are observed by FESEM are found to be about 5 µm thick and inclined at about 80 ° to the plane of the Si substrate. The Parylene-C µFTF was subsequently held between two appropriately spaced grips of a tension clamp and subjected to cyclic loading in a dynamic mechanical analyzer. Stress-strain measurements on the films allowed the determination of thestorage and loss moduli. Hence, the absorption coefficient α of the Parylene-C µFTFs is a function of temperature in the range 25 °C to 50 °C and the frequency in the low audible range up to 200 Hz. The acoustic absorption coefficient determined in this study is in a direction making about 80 ° to the microfiber axis. The dependence of α on temperature is found to be somewhat weak, whereas a much stronger dependence of α on frequency is observed. Three resonant frequencies have been determined for α, with the strongest resonance frequency located at about 125 Hz. This resonance is attributed to the resonant coupling between the incident sound waves and vibrating microfibers. We note that the absorption coefficient measured in these experiments is for acoustics waves incident in a transverse direction to the microfibrous columns. The film geometry is not amenable to applying this dynamic loading technique to directions closer to the column axis.

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