Temperature-dependent dynamic moduli of Parylene-C columnar microfibrinous thin films

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Abstract

Columnar microfibrinous thin films (μ F'TFs) of the polymer Parylene C with different microfiber inclination angles were fabricated using a physicochemical vapor deposition process whereby a collimated flux of hot monomers is obliquely directed towards a planar substrate. Each μ F'TF was then subjected to cyclic elastic loading in one of two orthogonal directions lying wholly in the substrate plane: either (i) normal or (ii) parallel to the morphologically significant plane of the μ F'TF. Dynamic storage and loss moduli were determined in the 1 to 80 Hz frequency range for temperatures between -40°C and 125°C . For all the columnar μ F'TFs, the storage and loss moduli for normal loading did not exceed their counterparts for parallel loading. All the columnar μ F'TFs were found to be softer than a bulk film of Parylene C. In both bulk and columnar forms, Parylene C was found to be rheologically not simple.

Keywords: Parylene C, dynamic mechanical analysis, storage modulus, loss modulus, microfibrinous film

1. Introduction

Thin films have been researched as optical, electrical, magnetic, chemical, mechanical, and thermal coatings for over 150 years [1]. These films range in thickness from a few angstroms (even molecular monolayers) to hundreds of micrometers, depending

5 on the specific application. They are made of a wide variety of raw materials including metals, glasses, and polymers [2].

Parylene C is a polymer with long-standing industrial use for electrically insulating coatings [3] and moisture barriers [4]. Bulk films of Parylene C are fabricated on substrates using a physicochemical vapor deposition process devised originally by Gorham
10 [5]. Free of pin holes, these films are used as interconnects [6, 7] and in microelectro-mechanical systems [8]. They are known to withstand temperatures as high as 220 °C and are resistant to most solvents [3, 9]. Accordingly, they are used as biomedical substrates [4, 10, 11, 12, 13].

Microfibrinous thin films (μ FTFs) of Parylene C can be fabricated on substrates by a
15 modification of the now-standard Gorham process [14, 15]. An μ FTF is porous, as it is an assembly of parallel microfibers of diameter $\sim 5 \mu\text{m}$. The shape of the microfibers can be upright circular-cylindrical, slanted circular-cylindrical, chevronic, helical, etc. The spacing between neighboring microfibers on a smooth substrate is $\sim 3 \mu\text{m}$, but almost any larger spacing between the microfibers can be obtained by pre-patterning
20 the substrate [15]. The potential of these μ FTFs for tissue growth [16] as for protein assays [17] has been experimentally established, and theoretical analysis indicates their promise for ultrasonic filters as well [18].

A μ FTF comprising microfibers of circular-cylindrical shape, whether upright or slanted with respect to the substrate plane, is called a columnar μ FTF. All microfibers
25 are inclined at an angle $\chi \in (0^\circ, 90^\circ]$ with respect to the substrate plane. We recently characterized the molecular and microstructural differences between columnar μ FTFs and bulk films of Parylene C [19]. Whereas bulks films are 83% crystalline and have only one crystal plane, the columnar μ FTFs have 55–68% crystallinity and four crystal planes. However, the static contact angles of water droplets on columnar μ FTFs were
30 similar to the static contact angles of water droplets on the bulk films [19].

Biological cells growing on a substrate subject the latter to contractile stress [20, 21]. Likewise, service as biomedical assays and ultrasonic filters will be stressful. Parylene-C μ FTFs also show promise as multifunctional materials [22, 23]. Therefore, although comprehensive knowledge of the mechanical properties of μ FTFs of
35 Parylene C would be useful for these applications, only a preliminary report on just

Figure 1: (a) Schematic of the process to deposit columnar μ FTFs of Parylene C. (b) Planar view of μ FTF on Si substrate in xz and yz planes. The angle χ_v of the collimated monomer flux with respect to the plane of the Si substrate and the microfiber inclination angle χ are marked. For each value of χ_v chosen, one substrate was aligned as shown in (c) and the other as shown in (d) during deposition.

one columnar μ FTF has been published as yet [24]. Moreover, for day-to-day applications, these properties should be determined as functions of both temperature and frequency.

In this paper, we report our experimental investigation of the frequency- and temperature-dependent storage and loss moduli of columnar μ FTFs of Parylene C (grown on planar substrates) subjected to cyclic elastic loading in one of two orthogonal directions lying wholly in the substrate plane: either (i) normal or (ii) parallel to the morphologically significant plane (MSP) of the μ FTF. The storage and loss moduli for normal loading are denoted by $E'_{\perp}$ and $E''_{\perp}$, while those for parallel loading are denoted by $E'_{\parallel}$ and $E''_{\parallel}$. We determine the dependences of all the measured moduli on the microfiber inclination angle χ .

The plan of this paper is as follows. In Sec. 2.1 we describe the procedure for fabricating the columnar μ FTFs of Parylene C. Next, in Sec. 2.3 we describe the mechanical experiments to determine $E'_{\perp,\parallel}$ and $E''_{\perp,\parallel}$. The measured data are presented and discussed in Sec. 3.

Figure 2: (a) Normal loading of a columnar μ FTF and (b) parallel loading of a columnar μ FTF, both fabricated with the same value of χ_v .

2. Materials and Methods

2.1. Fabrication of columnar μ FTFs

A $2.5 \times 1.3 \text{ cm}^2$ substrate of silicon (Si) was cleaned first for 10 min with acetone, then for 10 min with deionized (DI) water, then for 10 min isopropyl alcohol, and finally for 10 min with DI water. The substrate was then attached to a platform mounted on a motor assembly placed inside the *large chamber* of the PDS 2010 Labcoater (Specialty Coatings and Systems, Indianapolis, IN, USA), as shown schematically in Fig. 1(a). The shaft of the *rocking motor* in the assembly was oriented so that, during deposition, a collimated monomer flux of Parylene C would be obliquely incident on the substrate at an angle $\chi_v \in (0^\circ, 90^\circ]$ with respect to the substrate plane, as shown in Fig. 1(b). To fabricate the columnar μ FTF, 4 g of solid Parylene-C dimer (Specialty Coatings and Systems, Indianapolis, IN, USA) contained in a semi-cylindrical aluminum boat was placed in the *vaporizer* of PDS 2010 Labcoater. The chamber was then pumped down to 28 mTorr. In an automated process, the solid dimer was sublimated to gaseous form in the vaporizer held at 175°C . This vapor of dimers was passed into a furnace, held at 690°C , where it was dissociated into monomers [5]. This monomer flux was collimated by passage through a pipe and released through a nozzle towards the Si substrate. The monomers condensed on the substrate to form an assembly of slanted microfibers.

Let the MSP of a columnar μ FTF, which is the plane defined by the direction of the collimated monomer flux and the direction of inclination of the microfibers, be denoted

Figure 3: Cross-sectional SEM images of the MSPs of columnar μ FTFs fabricated with (a) $\chi_v = 30^\circ$, (b) $\chi_v = 52.5^\circ$, (c) $\chi_v = 67.5^\circ$, and (d) $\chi_v = 80^\circ$.

as the xz plane, while the substrate plane be denoted as the xy plane. In a predecessor study on columnar μ FTFs of Parylene C grown with different χ_v , the dependence of χ on χ_v was classified into four regimes as follows: χ varies very weakly with $\chi_v \in (30^\circ, 45^\circ)$, increases linearly with $\chi_v \in (45^\circ, 58^\circ)$, varies very weakly with $\chi_v \in (58^\circ, 75^\circ)$, and again increases linearly with $\chi_v \in (75^\circ, 90^\circ)$ [19, Fig. 4b]. The X-ray diffraction (XRD) spectrums of all columnar μ FTFs in each regime were found to be identical [19, Fig. 7]. Hence for this study, two columnar μ FTFs each were fabricated with $\chi_v = 30^\circ$, $\chi_v = 52.5^\circ$, $\chi_v = 67.5^\circ$, and $\chi_v = 80^\circ$, i.e., one in each of the four regimes. For each chosen value of χ_v , the substrate was aligned (i) as shown in Fig. 1(c) for one sample and (ii) as shown in Fig. 1(d) for one sample. In addition, one columnar μ FTF was grown with $\chi_v = 90^\circ$.

A bulk film was also deposited over a Si substrate but in the *reduced capacity chamber* of the PDS 2010 Labcoater, using 24 g of Parylene-C dimer. For this deposition, the motor assembly was not used and the nozzle was substituted by a baffle.

2.2. Cross-sectional imaging

Cross-sectional images of the MSPs of all columnar μ FTFs were taken using a scanning-electron microscope (SEM) [Model LEO 1530, Carl Zeiss, Darmstadt, Ger-

Figure 4: (a) $E'_{\parallel}$, (b) $E'_{\perp}$, (c) $E''_{\parallel}$, and (d) $E''_{\perp}$ at different temperatures for columnar μ FTF of $\chi_v = 30^\circ$. Numbers in the legend indicate temperatures.

many]. Each sample was first cleaved manually along the xz plane and sputtered with
 90 iridium in a sputter coater [Model K575X, Emitech, Fall River, MA, USA] on that plane
 for 28 s with 40 mA current. The SEM imaging software was used to measure χ with
 an accuracy of $\pm 0.1^\circ$.

2.3. Mechanical testing

The storage modulus is commonly determined by using profilometry data in the
 95 Stoney equation [25]. But this approach is limited to the measurement of the storage
 modulus of a homogeneous film in the thickness direction. Indentation methods using
 an atomic force microscope can only measure the local stiffness of the surface in the
 thickness direction [26]. Traditional optical methods using lasers [27], digital image
 correlation [28], and photoacoustic spectroscopy [29] are limited to room-temperature
 100 measurements, and the currently emerging moiré contouring technique's application
 for reliable measurements is time consuming [30]. Furthermore, those techniques
 which are capable of measurements at different temperatures require tedious instru-
 mentation and fabrication procedures [31].

Figure 5: (a) $E'_{||}$, (b) $E'_{\perp}$, (c) $E''_{||}$, and (d) $E''_{\perp}$ at different temperatures for columnar μ FTF of $\chi_v = 52.5^\circ$. Numbers in the legend indicate temperatures.

We therefore decided to use a Dynamic Mechanical Analyzer (DMA) [Model Q800,
 105 TA Instruments, Newcastle, DE, USA] to measure $E'_{\perp,||}$ and $E''_{\perp,||}$ over a range of frequencies and temperatures. In a preliminary study on a columnar μ FTF grown with $\chi_v = 45^\circ$ [24] we had measured $E'_{||}$ and $E''_{||}$ using DMA, with the μ FTF subjected to cyclic loads with a constant amplitude of deformation within the elastic regime at different frequencies f . For small deformations, i.e., in a linear material, the stress
 110 $\sigma = \sigma_o \sin(2\pi ft)$ while the strain $\varepsilon = \varepsilon_o \sin(2\pi ft + \delta)$ has a phase lag δ ; here, σ_o and ε_o are the respective amplitudes of stress and strain, and t is time. Then, $E' = (\sigma_o/\varepsilon_o) \cos \delta$ and $E'' = (\sigma_o/\varepsilon_o) \sin \delta$ [32, Chap. 3]. The viability of using the DMA for columnar μ FTFs was established in the preliminary study [24], the measured value of the storage and loss moduli being different from their counterparts reported
 115 [9] for bulk films of Parylene C.

To determine $E'_{||, \perp}$ and $E''_{||, \perp}$ of both columnar μ FTFs grown with the same value of χ_v per Figs. 1(c) and (d), the films were removed from the Si substrates with a razor moving along the y and the x directions, respectively. This was done to avoid curling of the columnar μ FTF in the loading direction. After this, the columnar μ FTFs

Figure 6: (a) $E'_{\parallel}$, (b) $E'_{\perp}$, (c) $E''_{\parallel}$, and (d) $E''_{\perp}$ at different temperatures for columnar μ FTF of $\chi_v = 67.5^\circ$. Numbers in the legend indicate temperatures.

120 were cut into rectangular shapes using glass slides and a razor, with the lengths of the samples lying between 5.84 mm and 7.37 mm and the widths between 4.33 mm and 6.38 mm. The thickness and the width of each columnar μ FTF were measured using digital vernier calipers. The lengths and the widths were chosen for each sample after initial experimentation with a room-temperature frequency sweep, in order to avoid the
 125 combined resonances of the sample and the DMA.

An initial strain-sweep test was performed at room temperature on all 10 samples to ensure that the amplitude of displacement (10 μ m) lay within the linear viscoelastic regime [32], *i.e.* $E'_{\parallel,\perp}$ were independent of the applied strain. First, ‘dynamic’ and ‘force’ calibrations were performed on the DMA. Next, the tension-film clamp was
 130 calibrated for ‘length’ and ‘stiffness’, per protocol. The sample was loaded in the tension-film clamp and the length of the sample was read from the instrument screen. Values of the sample’s width and thickness were fed to the ‘Q800 DMA’ software. The ‘Temperature Step - Sweep Frequency’ option in the ‘Multi-frequency Strain’ module was used. The temperature was set to vary from -40°C to 125°C in steps of 5°C .
 135 Before starting the measurement at a specific temperature, a wait time of 30 s was fixed

Figure 7: (a) $E'_{||}$, (b) $E'_{\perp}$, (c) $E''_{||}$, and (d) $E''_{\perp}$ at different temperatures for columnar μ F_{TF} of $\chi_v = 80^\circ$. Numbers in the legend indicate the temperatures.

in order for thermal equilibrium to be attained. At each temperature, a displacement amplitude of 10 μm was chosen for 20 linearly interpolated frequencies from 1 Hz to 80 Hz. At each frequency, the amplitude was stabilized for 4 cycles, and then the force and displacement data for that frequency were collected over 3 cycles. Based on the force and displacement measured by the DMA and the dimensions of the sample, $E'_{||,\perp}$ were calculated by the ‘Q800 DMA’ software.

3. Results and Discussion

The cross-sectional SEM images of the MSPs of four columnar μ F_{TF}s ($\chi_v < 90^\circ$) are shown in Fig. 3. These images clearly show that each columnar μ F_{TF} is an assembly of parallel microfibers, as in previous papers [19, 24].

The measured values of $E'_{||,\perp}$ of the columnar μ F_{TF}s with $\chi_v \in \{30^\circ, 52.5^\circ, 67.5^\circ, 80^\circ\}$ at different temperatures are shown in Figs. 4–7. For the columnar μ F_{TF} fabricated with $\chi_v = 90^\circ$ and the bulk film, $E'_{||} = E'_{\perp} = E''$ are similarly plotted in Fig. 8.

Figure 8: E' of (a) the columnar μ FTF deposited with $\chi_v = 90^\circ$ and (b) the bulk film. E'' of (c) the columnar μ FTF deposited with $\chi_v = 90^\circ$ and (d) the bulk film. Numbers in the legend indicate temperatures.

Figure 9: Comparison of $E'_{||}$ and $E'_{\perp}$ of columnar μ FTFs at temperatures (a) -40°C , (b) 15°C , (c) 70°C , and (d) 125°C . Solid lines represent $E'_{||}$, and dashed lines $E'_{\perp}$. Red, blue, green, pink, and black curves represent columnar μ FTFs fabricated with $\chi_v = 30^\circ, 52.5^\circ, 67.5^\circ, 80^\circ$ and 90° , respectively.

Let us note that the reported values of E' of bulk films of Parylene C are 2.76 GPa [9] and between 2.99 GPa and 3.84 GPa for bulk films grown in different vacuum conditions [33]. We found $E' = 2.15$ GPa for our bulk film at 1 Hz at room temperature, possibly due to different loading and deposition conditions.

For every columnar μ FTF made by setting $\chi_v < 90^\circ$, we found that $E'_{\parallel} > E'_{\perp}$, i.e., the columnar μ FTFs are stiffer in the xz plane (MSP) than in the yz plane (orthogonal to MSP). The excess of $E'_{\parallel}$ over $E'_{\perp}$ indicates that each microfiber tends to slide along its neighboring microfibers more during parallel loading than during normal loading, and that there is higher inter-microfiber friction in the MSP than in the yz plane.

In the chosen frequency range, $E'_{\parallel}$ increases weakly with frequency for all columnar μ FTFs at all temperatures, unlike the bulk film whose E' slightly decreases with increase in frequency. More importantly, both $E'_{\parallel,\perp}$ and $E''_{\parallel,\perp}$ for all columnar μ FTFs ($\chi_v \in \{30^\circ, 52.5^\circ, 67.5^\circ, 80^\circ, 90^\circ\}$) are at least 20 times lower than E' and E'' , respectively, of the bulk film. Accordingly, the columnar μ FTFs are much softer than the bulk film. This observation can be explained in part by the fact that amorphous materials have a weak atomic bonding (and, hence, smaller storage moduli) compared to crystalline materials. The columnar μ FTFs were found to have lower crystallinity (from 55% to 68%, depending on χ_v) than the bulk film (83%), as determined from XRD analysis [19]. Furthermore, the columnar μ FTFs have four crystal planes, whereas the bulk film has only one crystal plane [19]. More grain boundaries in a columnar μ FTF should soften the material. Next, the cross-sectional diameter of a microfiber is $\sim 4.5 \mu\text{m}$ and the center-to-center distance between neighboring microfibers is $\sim 7.5 \mu\text{m}$. Hence, we estimate the porosity of a columnar μ FTF to be $\sim 70\%$. Moreover, the microfibers contain nanopores due to the physicochemical nature of the deposition process [14, 34]. Thus, the porosities at the microscale and the nanoscale, the grain boundaries, and the amorphous nature of the columnar μ FTFs account for their lower storage and loss moduli in comparison to those of the bulk film.

The low values of $E'_{\parallel,\perp}$ of the columnar μ FTFs suggest their applicability for microfluidics [35]. Despite being softer than the bulk film of Parylene C, at room temperature the columnar μ FTFs are stiffer than bulk poly-dimethyl siloxane (PDMS), which has an (static) elastic modulus of 20 MPa [36] and is commonly used in mi-

Figure 10: Comparison of $\tan \delta_{||}$ and $\tan \delta_{\perp}$ of columnar μ FTFs at (a) 9.3 Hz, (b) 30.1 Hz, (c) 50.9 Hz, and (d) 71.7 Hz. Solid lines represents $\tan \delta_{||}$ and dashed lines $\tan \delta_{\perp}$. Red, blue, green, and pink curves represent columnar μ FTFs fabricated with $\chi_v = 30^\circ$, 52.5° , 67.5° , and 80° , respectively.

crofluidics [37]. Since the $E'_{||,\perp}$ of the columnar μ FTFs of Parylene C lie in the MPa regime, they are also capable as synthetic scaffolds [16, 38].

An examination of the values of $E'_{||,\perp}$ of all columnar μ FTFs at four different temperatures (-40°C , 15°C , 70°C , and 125°C) in Fig. 9 suggests that $E'_{||,\perp}$ are nonlinearly dependent on χ_v and that the columnar μ FTF fabricated with $\chi_v = 90^\circ$ has the highest $E'_{||,\perp}$. Furthermore, we note that as χ_v increases, the difference $E'_{||} - E'_{\perp}$ decreases at all temperatures.

For every columnar μ FTF made by setting $\chi_v < 90^\circ$, we found that $E''_{||} > E''_{\perp}$. But their values are not very different in Figs. 4 to 8, and are actually equal for $\chi_v = 90^\circ$. Furthermore, $E''_{||,\perp}$ increase with χ_v . As χ_v increases, the inter-microfiber spacing decreases and hence the contact friction between neighboring microfibers increases during loading, leading to higher $E''_{||,\perp}$.

We observe two minor peaks in the curves of $E''_{||}$ and $E''_{\perp}$ as functions of frequency. We call them minor peaks because the increments in $E''_{||}$ and $E''_{\perp}$ are less than 2 MPa and both $E'_{||}$ and $E'_{\perp}$ have smooth variations. We suspect each of these peaks is due to a combined resonance of the sample and instrument. Although such peaks are known

to occur at loading frequencies above 100 Hz for different materials, the low masses of our samples are causing the minor resonances to be observed below 100 Hz.

Examining Fig. 10 for $\tan \delta_{\parallel} = E''_{\parallel}/E'_{\parallel}$ and $\tan \delta_{\perp} = E''_{\perp}/E'_{\perp}$ as functions of temperature, we note that all columnar μ FTF have two peaks, similar to many bulk polymers [32, Sec. 7.3.1]. The lower-temperature-peak for four different frequencies of cyclic loading was found to lie either at -5°C or 0°C . The higher-temperature peak (averaged over the four frequencies of cyclic loading) was found to lie between 85°C and 95°C for parallel loading, and between 90°C and 105°C for normal loading. Thus, there is a variation of 10°C for the higher-temperature peak identified with the type of loading, frequency of loading, and also among the columnar μ FTFs grown with different values of χ_v . Whether or not the higher-temperature peak can be designated as the glass transition temperature T_g requires a shear test [39], but that turned out to be destructive. Further, we found the temperature peaks of bulk film to be the same as columnar μ FTFs based on similar plots as Fig. 10, but for bulk film. Published values of T_g lie between 30°C and 60°C for $20\text{ }\mu\text{m}$ -thick bulk films [33], while one report has $T_g = 92^{\circ}\text{C}$ for $5\text{ }\mu\text{m}$ -thick bulk films [40] and another report has $T_g = 90^{\circ}\text{C}$ for $5.8\text{ }\mu\text{m}$ -thick bulk films [41].

Time-Temperature Superposition (TTS) [42] was attempted over a narrow range of frequencies, 10 Hz to 50 Hz, where $E'_{\parallel,\perp}$ vary linearly with frequency for all the columnar μ FTFs and the bulk film. But smooth master curves for $E'_{\parallel,\perp}$ with respect to frequency could not be constructed for any of the ten samples, which implies that neither the columnar μ FTFs nor the bulk film of Parylene C are rheologically simple. This conclusion is in agreement with the literature that crystalline polymers are in general not rheologically simple [32, p. 251]. Both the bulk film and the columnar μ FTFs are crystalline, the former having a higher crystallinity than the columnar μ FTFs [19]. Indeed, the constructed TTS curve for any of the nine columnar μ FTFs was found have less deviation from a theoretically smooth master curve than the constructed TTS curve for the bulk film.

225 4. Conclusion

We note that for all the columnar microfibrinous films, independent of the angle χ_v of the collimated monomer flux with respect to the substrate plane, the storage modulus in the morphologically significant plane is higher than in the vertical plane orthogonal to the MSP. All of the columnar μ FTFs have storage and loss moduli lower
230 than those of the bulk film. The temperature peaks in the loss moduli of the columnar μ FTFs and bulk film were found to lie between 85 °C and 105 °C. Parylene C in the bulk as well as the microfibrinous forms was found to be rheologically not simple. A knowledge of these properties will help in designing MEMS, towards the larger goal of developing microfibrinous multifunctional metamaterials of Parylene C [23] and for
235 biomedical applications in particular.

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Figure Captions

Fig. 1: (a) Schematic of the process to deposit columnar μ FTFs of Parylene C. (b) Planar view of μ FTF on Si substrate in xz and yz planes. The angle χ_v of the collimated monomer flux with respect to the plane of the Si substrate and the microfiber inclination angle χ are marked. For each value of χ_v chosen, one substrate was aligned as shown in (c) and the other as shown in (d) during deposition.

Fig. 2: (a) Normal loading of a columnar μ FTF and (b) parallel loading of a columnar μ FTF, both fabricated with the same value of χ_v .

Fig. 3: Cross-sectional SEM images of the MSPs of columnar μ FTFs fabricated with (a) $\chi_v = 30^\circ$, (b) $\chi_v = 52.5^\circ$, (c) $\chi_v = 67.5^\circ$, and (d) $\chi_v = 80^\circ$.

Fig. 4: (a) $E'_{\parallel}$, (b) $E'_{\perp}$, (c) $E''_{\parallel}$, and (d) $E''_{\perp}$ at different temperatures for columnar μ FTF of $\chi_v = 30^\circ$. Numbers in the legend indicate temperatures.

Fig. 5: (a) $E'_{\parallel}$, (b) $E'_{\perp}$, (c) $E''_{\parallel}$, and (d) $E''_{\perp}$ at different temperatures for columnar μ FTF of $\chi_v = 52.5^\circ$. Numbers in the legend indicate temperatures.

Fig. 6: (a) $E'_{\parallel}$, (b) $E'_{\perp}$, (c) $E''_{\parallel}$, and (d) $E''_{\perp}$ at different temperatures for columnar μ FTF of $\chi_v = 67.5^\circ$. Numbers in the legend indicate temperatures.

Fig. 7: (a) $E'_{\parallel}$, (b) $E'_{\perp}$, (c) $E''_{\parallel}$, and (d) $E''_{\perp}$ at different temperatures for columnar μ FTF of $\chi_v = 80^\circ$. Numbers in the legend indicate temperatures.

Fig. 8: E' of (a) the columnar μ FTF deposited with $\chi_v = 90^\circ$ and (b) the bulk film. E'' of (c) the columnar μ FTF deposited with $\chi_v = 90^\circ$ and (d) the bulk film. Numbers in the legend indicate temperatures.

Fig. 9: Comparison of $E'_{\parallel}$ and $E'_{\perp}$ of columnar μ FTFs at temperatures (a) -40°C , (b) 15°C , (c) 70°C , and (d) 125°C . Solid lines represent $E'_{\parallel}$, and dashed lines $E'_{\perp}$.

Red, blue, green, pink, and black curves represent columnar μ FTFs fabricated with $\chi_v = 30^\circ, 52.5^\circ, 67.5^\circ, 80^\circ$ and 90° , respectively.

415 Fig. 10: Comparison of $\tan \delta_{\parallel}$ and $\tan \delta_{\perp}$ of columnar μ FTFs at (a) 9.3 Hz, (b) 30.1 Hz, (c) 50.9 Hz, and (d) 71.7 Hz. Solid lines represents $\tan \delta_{\parallel}$ and dashed lines $\tan \delta_{\perp}$. Red, blue, green, and pink curves represent columnar μ FTFs fabricated with $\chi_v = 30^\circ, 52.5^\circ, 67.5^\circ$, and 80° , respectively.

Fig. 1

Fig. 2

(a)

(b)

(a)

(b)

(c)

(d)

Fig. 4

Fig. 5

Fig. 6

Fig. 7

Fig. 8

Fig. 9

Fig. 10