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Research Paper

Robust mechanical property measurements of fibrous parylene-C thin-film substrate via moiré contouring technology

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ABSTRACT

Parylene-C is a bio-inert, bio-compatible and relatively inexpensive material with many bio-medical applications from coatings for implantable devices to bio-scaffolds. The main objective of this research was to demonstrate a novel approach to accurately measure the mechanical properties of free-standing fibrous thin-film substrates (TFS) of parylene-C. For that purpose, a two-stage experimental protocol based on the use of moiré contouring technology was developed. In this protocol, local measurements employing an advanced moiré setup that uses non-conventional illumination (i.e. evanescent field) are first performed to gather high-resolution information on a small region of the specimen; then, global measurements based on shadow moiré are performed to monitor the overall behavior of the membrane. The protocol was first calibrated for an aluminum foil and then partially applied to the fibrous parylene-C TFS. Material properties extracted from experiments are f0ully consistent with the data reported in literature and the results of a hybrid identification procedure based on the combination of finite element analysis and nonlinear optimization. The results will help lay the foundation for developing a comprehensive understanding of the influence that morphology and stresses play in the ability to enhance and sustain cell growth and tissue development, for biomedical applications.

1. Introduction

Approved by the US Food and Drug Administration in the 1970s, parylene-C is a bio-inert, bio-compatible, and relatively inexpensive material to conformally coat various surfaces. Much research has been carried out on its use for external (Cieślik et al., 2012; Pierstorff et al., 2008) as well as implantable (Schmidt et al., 1988; Tooker et al., 2005; Westedt et al., 2006) medical devices. Fundamental studies on the attachment, growth, and proliferation of cells on dense films

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of parylene-C have shown promise for their use as substrates for tissue growth (Chang et al., 2007; Song et al., 2009; Tan and Craighead, 2010).

Dense films of parylene-C have been grown by physicochemical vapor deposition for about 5 decades (Gorham, 1966). A few years ago, a hybrid technique combining physicochemical vapor deposition with oblique angle deposition (Holland, 1953)—used commonly to grow sculptured thin films (Lakhtakia and Messier, 2005) of inorganic materials and metals—was devised (Pursel et al., 2005) to grow parylene-C thin films endowed with a volumetric fibrous morphology. The fibers are nominally parallel to each other and can have certain shapes such as tilted columnar, chevronic, and helical. The surfaces of these fibrous thin films exhibit a thickness-dependent hydrophobicity (Wei et al., 2010a). Experiments with fibroblast cells strongly indicate their promise as substrates for the attachment, growth, and proliferation of cells (Demirel et al., 2007; Wei et al., 2010b, 2012). In addition, fibrous thin-film substrates (TFS) of parylene-C can be free-standing (Wei et al., 2012)—which is very desirable for biomedical applications.

While there is tremendous potential for these free-standing fibrous TFS of parylene-C in biomedical applications (Wei and Lakhtakia, 2012a), very little is known about their mechanical properties, particularly when considering long-term integrity. Cellular attachment has been shown to generate high stresses, causing detachment and rupture (Delanoë-Ayari et al., 2011; Mello et al., 2007). But the experimental determination of the mechanical properties of thin films poses difficulties relating to sample handling, sample alignment, accurate measurement of strain, and accurate measurement of applied force. Therefore, biomedical researchers simply measure geometrical attributes when investigating cell attachment and growth. Dense films of parylene-C have been mechanically characterized with a traditional macroscopic setup (Song et al., 2009), but that arrangement will not work for films with volumetric fibrous morphology. Nano-indentation, a broadly used technique to measure mechanical properties of dense thin films (Fischer-Cripps, 2006; Bhushan, 2007), is not suitable either because it can damage films with volumetric fibrous morphology (Seto et al., 1999). Furthermore, the nano-indentation test is not a direct measurement of mechanical properties, and an empirical model has to be developed to extract the elastic modulus, yield stress, strain hardening coefficient, residual stress, and fracture toughness from the recorded indentation load–depth curve.

A method that is extensively used to mechanically characterize thin films is the bulge test (Wineman et al., 1979; Small and Nix, 1992; Paul and Gaspar, 2008). This test simplifies many of the problems that other methods pose, but classical interferometry is inadequate for the accurate measurement of the deflection of a thin film with high compliance. One flexible approach that can accommodate a large range of deflections (critical for thin film measurements) is moiré contouring (Sciammarella and Sciammarella, 2012). Utilization of moiré methodologies makes it possible to measure deflections in the sub-micrometer and micrometer range. In a new experimental protocol devised by us, two different moiré methodologies are utilized to obtain the mechanical properties of a thin film. First, local measurements on a small region of the thin film are performed by utilizing a novel form of projection moiré based on un-conventional illumination. Second, classical shadow moiré is used to obtain full-field measurements of the thin film. Local and global measurements should provide similar results (i.e. measured displacement must be of the same order of magnitude), and can therefore be used as checks on each other.

Moiré methodologies appear to be very applicable for the mechanical characterization of fibrous TFS of parylene-C. For this purpose, a free-standing fibrous TFS with chevronic morphology was fabricated following the procedure outlined in (Wei et al., 2012). A series of global measurements were performed on the free-standing fibrous TFS for various loading conditions. Due to the geometric configuration of the testing apparatus, local measurements on the TFS were not possible at the time. But the out-of-plane displacement field gathered from global measurements was compared with the results of a finite element model that was used to reproduce the inflation experiment that was carried out in the lab. The difference between experimental data and numerical results was expressed by an error function depending on the mechanical properties of the tested material. The in-plane mechanical properties of the fibrous parylene-C TFS determined via optimization are quite reasonable when compared with those of dense films reported in the literature (2012, 〈http://www.mit.edu/~6.777/matprops/parylene.htm〉).

2. Moiré contouring of thin films

Projection moiré is a contouring technique used to get the shape of an object by projecting a grating onto its surface. The profile of the surface modulates the frequency of the grating. From the modulation information, the profile of the surface can be obtained. The retrieval can be done either by combining optics and computations or by applying only numerical procedures. In the latter case, one can refer to digital moiré (Sciammarella and Sciammarella, 2012). Local measurements allow us to gather information for a limited region of the specimen which might experience the largest displacement or was more likely to host mechanical failures. A novel form of moiré contouring utilizes a diffraction grating illuminated by light beams inclined beyond the limit angle that produces total internal reflection (Sciammarella et al., 2010). This generates an evanescent field (Toraldo di Francia, 1958; Born and Wolf, 2002) that greatly increases the level of resolution available for the experiments. Moreover, near-field phenomena can then be detected using a standard far-field optical microscope. This method was successfully utilized (Sciammarella et al., 2009) to observe nano-crystals reaching ±5 nm accuracy, and images of nano-crystals were recorded with a standard optical microscope. Global measurements are done using classical shadow moiré (Sciammarella and Sciammarella, 2012). Both global and local measurements were carried out on an aluminum foil in order to calibrate the whole process by testing a known material. Since the evanescent field rapidly decays as it travels from the grating to the specimen surface, it is necessary to put the grating very close to the specimen surface. This requirement could not be satisfied in the case of the parylene-C TFS specimen as the clamping device for the Parylene-C TFS required a depth that made it impossible to get the grating close enough to the membrane. To achieve this condition, a considerable redesign of the system was required.