NEW NCMC Focused Project: *High-Throughput Modulus Measurements of Hydrated Polymer Gels*

The NIST Combinatorial Methods Center (NCMC) is pleased to announce a new focused project on the modulus of hydrated polymer gels. This project will adapt NCMC buckling-based techniques[1] to the quantitative measurement of the modulus of soft, hydrated systems used in a variety of products, such as contact lenses. We are inviting NCMC member organizations and others to join this project. Focus Project members will help the NCMC set define instrument parameters, and help to develop and specify the model systems we will use to demonstrate of this exciting new technology. Research results will be available to focused project members throughout the project period and member organizations will be able to participate directly with NIST researchers in development of the new measurement tools.

Introduction

Soft polymer systems such as hydrogels present a considerable challenge to existing mechanical testing techniques. Additionally, the increasing pace of research and development of such products demands that measurements should occur in a high-throughput (HT) manner. In response to these challenges, this Focus Project aims to produce a platform for the HT measurement of the modulus of hydrated soft polymer materials and products. This includes:

- Design and prototyping of an appropriate support structure for mounting and handling arrays of multiple soft hydrogel specimens
- Design of a method to deposit sensor films onto individual elements of specimen arrays
- Design of a coordinated platform that integrates sample preparation, manipulation, and HT measurements of specimen modulus
- Demonstration of the platform through a validation study focused on a selected model specimen system under a set of relevant experimental parameters

[1] *CM Stafford, et al., Nature Materials 3, (2004).*

Technical Background

Hydrated polymer gels, or hydrogels, are soft materials capable of absorbing large quantities of water. Because they possess qualities very similar to those of soft tissues, they have found widespread use in the field of biomaterials with applications ranging from contact lenses to tissue engineering.

One of the most important considerations in the evaluation of hydrogels for biomedical applications is the elastic modulus. The elastic modulus relates to several important factors including flexibility, adhesion, swelling behavior, and the potential for cell proliferation and growth. Current methods for assessing the moduli of hydrogels include tensile and compression studies that are typically oriented towards the measurement of single specimens in a non-automated manner. Moreover, such techniques are generally geared towards stiff specimens (*e.g.*, structural polymers), and they do not always offer a means to measure systems that need to be conditioned by a controlled environment (*e.g.*, constant hydration).

We have developed a high-throughput metrology[1] for measuring the elastic modulus of thin films and coatings. This methodology leverages an elastic buckling instability that occurs upon compression of a stiff upper film supported by a soft elastic substrate. The periodicity of the buckling pattern is primarily dependent on the modulus ratio between the film and substrate as well as the thickness of the upper film:

$$\frac{E_f}{(1-\nu_f^2)} = \frac{3E_s}{(1-\nu_s^2)}\left(\frac{d}{2\pi h}\right)^3 \qquad (1)$$

where E is the elastic modulus, v is Poisson's ratio, h is the thickness of the film, and d is the wavelength of the instability (subscripts f and s denote the film and substrate, respectively). Poly(dimethylsiloxane) (PDMS, Sylgard 184, Dow Chemical) was chosen as the elastic foundation since it is optically transparent, it approximates an ideal elastomer, and its modulus

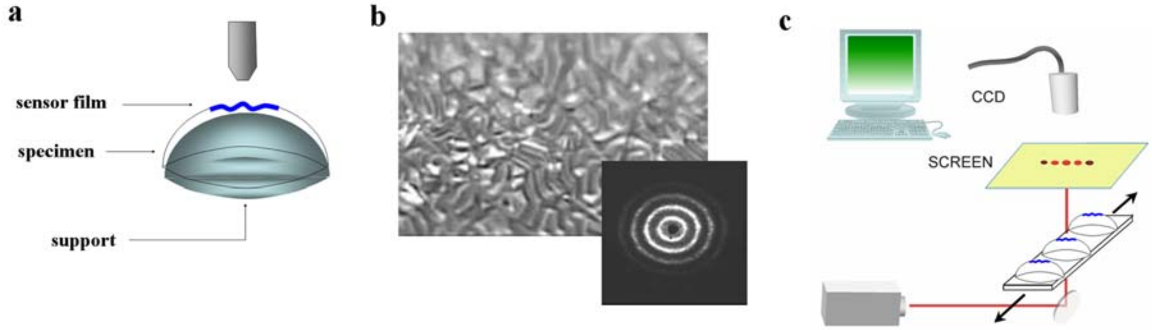

Figure 1: High Throughput Measurement Platform

can be tuned by the mixing ratio of base to curing agent ($E_s \approx$ (1 to 2) MPa). In our validation studies, the upper film was the unknown to be measured (all else being known or constant). We propose in this study to invert the experimental design by using a sensor film of known modulus and thickness (*e.g.*, PS, PMMA, Teflon AF), thus rearranging Eq. 1 as follows:

$$\frac{E_s}{(1-\nu_s^2)} = \frac{E_f}{3(1-\nu_f^2)}\left(\frac{2\pi h}{d}\right)^3 \qquad (2)$$

Here, the unknown to be determined is the modulus of the soft elastic substrate. The thickness of the sensor film is chosen such that the wavelength of the buckling instability can be measured by small angle light scattering (SALS), thus enabling high-throughput measurement of the substrate modulus.

Figure 1 illustrates our metrology scheme using a contact lens specimen as an example. Fig 1a shows the specimen geometry, where the sample is supported by a transparent hemispherical substrate. A sensor film, deposited on the sample, undergoes a buckling instability as our preliminary studies demonstrate (Fig 1b). The high-throughput measurement platform employs light scattering to ascertain the buckling wavelength (Fig 1c, Fig 1b-inset). This approach is amenable to the measurement of multiple specimen arrays in a rapid automated manner.

Using this scheme, we will construct a platform for measuring the modulus of hydrated polymer gel specimens in a combinatorial and high-throughput manner. This methodology will involve developing:

- A method for the preparation of multi-specimen libraries that includes means to hydrate samples and which is amenable to instrumented automation.
- Techniques for the rapid measurement of the modulus of specimens across these arrays, also amenable to automation.
- Device designs and measurement protocols for each of these previous points.
- A validation study aimed at demonstrating the quantitative measurement of modulus of model specimens.

We will work with Focus Project members to define appropriate model systems and specific parameters of interest such as specimen geometry (*e.g.*, contact lens), crosslink density, and/or degree of swelling that will demonstrate the robustness of the platform.

Project Deliverables

Total Project Period is 1.5 years (6 quarters)

Q1/Q2:

- Demonstration of quantitative, reproducible, modulus measurements of a model specimen via buckling scheme.
- Definition and transfer of appropriate model specimens for study.
- A prototype sample support structure for multiple specimens.
- Demonstration of automated measurement of specimen modulus via light scattering or optical microscopy.

Q3/Q4:

- Demonstration of a method for deposition of sensor films onto single specimens, that is amenable to automation.
- Launch of validation study on set of model specimens.
- Demonstration of a method for the deposition of sensor films onto multiple specimens.

Q5/Q6:

- Demonstration of coordinated platform: combination of multiple sample deposition scheme and high-throughput measurement scheme.
- Launch technology transfer to member and/or engineering firm proxy.
- Completion of validation study.
- Written device designs and method protocols supplied.
- Complete technology transfer to member and/or engineering firm proxy.

Collaboration and Project Dissemination

A discussion meeting will be arranged six weeks after the formal launch of the project as well as at six-month intervals for the duration of the project. NCMC will facilitate dissemination and communication among members of the focus project. Written reports will be sent to the members at six-month intervals, with updates more frequently via conference calls. In order to facilitate the collaboration, specifications for methods, instruments, programs, data analysis, and other aspects of this work will be available to members during the course of the project. A summary report will be provided within two months of the end of the project. The NCMC labs will be open to prearranged visits from member scientists interested in hands-on participation in method development and technology transfer.

As with base level membership in the NCMC, all of the research carried out in the Focused Project is non-proprietary and is intended for publication in the public domain. No proprietary information or materials will be solicited or accepted by NIST from member organizations. The scope of the work by NIST included in this focused project will be limited to milestones described in the Appendix A of the focused project agreement.

For more information

To learn more about the High-Throughput Modulus Measurements of Hydrated Polymer Gels Project:

Call: (301) 975-8526
Write: Combinatorial Methods Center
NIST
100 Bureau Drive, MS 8542
Gaithersburg, MD 20899-8542
e-mail: mfasolka@nist.gov
or visit the NCMC website at
http://www.nist.gov/combi

The Focused Project Agreement Form and Statement of Work (Appendix A) are available on the website for downloading (in .pdf format).