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Mapping micromechanical properties of soft polymer contact lenses

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ABSTRACT

We present comparative micromechanical characterization of several commercial soft silicone hydrogel contact lenses, which allows for the examination of spatial distribution of different regions with local mechanical properties within the lens under practical wet conditions. We employ elastic contact mechanic model and corresponding analysis of force–distance curves collected with high-resolution atomic force microscopy measurements performed within elastic deformation limits. The measurements were performed on the lens cross section to map the micromechanical properties distribution within the subsurface regions and bulk material of the different lens. In addition, we have studied topography and mechanical properties of the lens surfaces, which come into direct contact with the surface of the eye and eyelid. AFM images show high contrast distribution maps for the adhesive and mechanical properties of the different microstructured regions such as pores, lamellae and different material inclusions within the lenses. Additional indentation experiments allow for collection of quantitative data for micromechanical properties from different regions within the lens structure and correlate these data with lens-averaged macroscopic measurements available in the literature.

1. Introduction

The mechanical properties of silicone hydrogel contact lenses, which critically depend upon lens water content, porosity, and oxygen permittivity, are the most important parameters affecting their ultimate performance [1]. State of the art contact lens materials should be soft and flexible enough to provide comfort to the wearer and reduce complications associated with constant stress within the eye such as papillary conjunctivitis and changes in corneal curvature [2,3]. At the same time the lens material needs to be rigid enough to maintain overall shape and withstand multiple stress cycles in saline environments, while prolonging optical performance and clarity during extended wear [4].

The introduction of silicone hydrogel materials for lens fabrication has allowed for the design of new, high performance lenses. Because these silicone-based materials are intrinsically hydrophobic and have higher elastic moduli than previous generations of lens materials, much focus has been placed on increasing water content and oxygen transport in the lens and reducing surface stiffness for maximum comfort to the wearer [5–7]. For example, to reduce differences in surface chemistry between the lens material and the ocular surface while preventing tear film breakup, a surface coating can be applied or wetting agents can be incorporated into the bulk of the lens structure. Most popular current designs involve physical and chemical surface treatments to address these issues and modify lens–eye interactions including adhesion and lubrication [8]. Therefore, it becomes even more important to precisely characterize local mechanical properties distributions of these multiphase materials under relevant practical conditions in addition to traditional tensile testing.

Most current mechanical measurement methods involve conventional tensile testing to evaluate elastic modulus of the lens materials [9,10]. However these conventional tensile methods have several drawbacks. First, it is hard to compare the stiffness of different contact lenses as the measurements strongly depend on the sample preparation: such factors as small defects introduced during fabrication or clamping prior to testing and differences in thickness between lenses with different optical power dramatically reduce the precision. Second, the complex near semi-spherical sample shape makes measured values vary greatly; thus a simple flat slab shape is frequently used instead of actual lenses for tensile testing [11]. Furthermore, tensile tests are only able to provide an averaged elastic modulus of the entire lens, without any means to differentiate specific local properties from the lens components. In the case of surface modification of contact lenses the mechanical properties of the coating will dominate in the eye-lens contact interface although the overall rigidity is controlled by the bulk properties of lens matrices [12]. Thus high resolution, spatially

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capable techniques should be employed to evaluate distributed mechanical properties of multiphase lens materials.

The AFM-based surface force spectroscopy (SFS) technique is a well known approach for the characterization of micromechanical properties of multiphase polymeric materials with nanoscale resolution [13,14]. This technique was successfully used for micromechanical characterization of complex polymeric composite materials, grafted polymers, natural polymers, nanoscale polymer films, soft biomaterials and individual molecules [15–27]. AFM-based techniques have been widely used in vision science to study surface topography [28], friction [29], and protein absorption [30] for contact lens materials and eye tissue [17,31], however they are rarely used for probing surface mechanical properties of this class of materials due to the complexity of probing under wet conditions.

Several successful examples of measuring micromechanical properties of the surface layers of hydrogel contact lens have been published by Somorjai et al. [32,33]. In these studies, it was found that elastic modulus of the lens surface depends strongly on the loading rate and water content within the lens. However only thin surface regions were studied and the spatial distribution of mechanical properties across the lens surface and sub-surface regions was not examined in this study. To the best of our knowledge, no studies on the variations in mechanical properties of different structural regions within the soft contact lenses (exterior and interior) have been performed to date.

From an experimental prospective, such measurements can be performed in two probing manners: the fast, high-frequency force-tapping mode (under different common trade names such as Bruker's Quantitative Nanomechanics (QNM)) and by using standard surface force spectroscopy (SFS) in which a two dimensional array of force–distance curves (FDCs) is collected in static mode and analyzed [34]. FDCs collected in both ways contain tip sample interaction information and can be used to calculate sample mechanical properties using known tip parameters and different models of contact mechanics [35,36]. In the force-tapping mode, an AFM tip is driven sinusoidally at frequencies much lower than the cantilever's first resonant peak (typically 1 or 2 kHz) and briefly interacts with the sample surface in the middle of each cycle [37]. This measurement mode allows for the determination of various mechanical properties of the sample surface, such as elastic modulus and adhesion, as well as surface topography with high resolution in a short amount of time. However, as a result of the short interaction time, the mechanical measurements performed in this mode lack precision and usually only provide qualitative contrast maps of surface distribution of mechanical properties [14]. In SFS, the AFM tip moves in saw tooth-like motion indenting into the sample during each cycle. Due to the piezoelement mobility limitations, the frequency of these indentations are limited to several tens of Hz [38], however the well-defined FDCs enables the determination of quantitative data for the material with high precision [13].

In present study we suggest a practically-valuable methodology to characterize spatial distribution of mechanical properties within the soft microstructured materials of commercial contact lenses including both exterior and interior microscopic regions. Using commercially-available soft silicone hydrogel contact lens materials as an example we demonstrate that high frequency measurements to acquire high resolution maps of topography and mechanical properties can be combined with static FV nano-indentations to provide reliable micromechanical measurements to such complex composite materials under wet conditions. We mapped the micromechanical properties of the lens surface as well as lens cross-sections to reveal complex sub-surface morphology with greatly different elastic responses that cannot be obtained with conventional macroscopic mechanical testing.

2. Experimental

2.1. Contact lens preparation

In this work, four different commercially available silicone-hydrogel contact lens brands were selected for comparative studies: Balafilcon A (Purevision, Bausch & Lomb Inc.), Senofilcon A, (Acuvue Oasis, Johnson & Johnson Vision Care Inc.), Lotrafilcon B (AirOptix Aqua, CIBA Vision Corp.) and Comfilcon A (Biofinity, CooperVision Inc.) purchased through Contactlenses Ltd. Manufacturer reported values for several physical characteristics of these lenses are summarized in Table 1.

Lenses were measured immediately after removal from their original blister pack and discarded after one day of measurements. Each lens was sectioned into several pieces using steel razor blades under wet conditions to avoid any drying (Scheme 1, a). The lenses were attached to metal sample disks (Ted Pella Inc.) using double sided tape for AFM measurements on the lens surface (Scheme 1, b). The measurements were performed within a small droplet of saline solution (taken from the blister pack) covering the piece of the lens deposited onto the metal disc. For AFM measurements of the lens cross-sections, pieces of the lens were firmly locked between two metal plates with the freshly cut surface facing upwards (Scheme 1, c). Both metal plates were fully submerged in the liquid for the measurement. To find regions suitable for the AFM measurements, lens sections were examined with a high resolution microscope (BX51, Olympus) in the dry state followed by quick rehydration. Poly(dimethyl siloxane) (PDMS) was used as a control model substrate for elastic modulus measurements [39]. Centimeter-thick PDMS substrates were prepared using a Sylgard 184 Silicone Elastomer Kit (Dow Corning, USA). The elastomer base was vigorously mixed with curing agent in a 5:1 ratio (w:w) for 10 min, then placed under vacuum at room temperature for 30 min in order to remove all remaining air bubbles. The mixture was cured overnight at 70 °C and allowed to cool before use in AFM measurements.

2.2. AFM measurements

AFM and SFS measurements were performed with a Dimension Icon AFM microscope (Bruker). All measurements were performed with the lens submerged in the original solution from the lens package using a fluid cell. Soft aluminum coated AFM tips (Mikromasch) with nominal spring constants of 0.2 N/m were used for the measurements. Prior to each new sample measurement full tip characterization was conducted. Tip shape was estimated using the blind estimation method using a titanium roughness sample (Bruker) (see Fig. S1, Supporting Information). The blind estimation method allows for precise imaging of the AFM tip by scanning surfaces with sharp features [40]. The observed radius of curvature of the tips varied within the 10–30 nm range. Deflection sensitivity was determined by making FDCs on a sapphire crystal and the spring constant was calculated using the thermal calibration method.

Table 1
The characteristics of contact lenses used in this study.

Name	Manufacturer	Material	Water content[a], %	Surface treatment	Modulus, MPa[a]
Air Optix	Ciba Vision	Lotrafilcon B	33	Plasma coating	1.0
PureVision	Bausch and Lomb	Balafilcon A	36	Plasma oxidation	1.1
Acuvue Oasys	Johnson and Johnson	Senofilcon A	38	NA	0.72
Biofinity	CooperVision	Comfilcon A	48	NA	0.75

[a] Manufacturer reported values from Ref. [7].