SCREENING METHODS FOR WOVEN MATERIALS
AS ARTIFICIAL CARTILAGE

A Dissertation

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In the orthopedics industry, there exists a void in the continuum of care associated with joint pain due to cartilage damage and wear. Currently, patients are asked to endure joint pain with only over-the-counter pain medication to ease discomfort. When the pain becomes unbearable, joint surgery is performed. As the average age of joint replacement recipients decreases, the necessity of revision surgeries increases due to limited hardware life spans. Revision surgeries are more traumatic and require longer recovery times than primary replacements. Although some procedures, like unicondylar knee replacements and hip resurfacing, are less invasive and preserve more tissue than total joint procedures, a minimally invasive short-term cartilage replacement is desired. A synthetic plug of a three-dimensionally woven material has been forwarded as a possible solution to delay the need for a total joint replacement while effectively relieving pain. Once implanted, micro-motion between fibers within the device will be experienced during each loading cycle. The minimization of wear during these events is important not only to avoid device failure but also to minimize a biological response. In the current work, various screening methods for fiber materials are evaluated and range from simple mechanical property testing using an atomic force microscope (AFM),
direct fiber-on-fiber wear using a rig test, to pin-on-disk accelerated life wear testing. It is found that direct fiber-on-fiber wear tests provide distinguishable rankings of fiber materials according to wear coefficient and identify polyethylene terephthalate (PET) and ultra-high-molecular-weight polyethylene (UHMWPE) as candidates for further investigation. Pin-on-disk tests have allowed the wear mechanisms of three-dimensional fabrics to be explored and provide insight for further optimization of weave parameters and fiber materials.
I dedicate this to my parents, who have provided endless love and support, and to my beautiful wife, Caitlin, who has always claimed that I am her “held to maturity investment.”
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<tr>
<td>$A_i$</td>
<td>Cross-sectional area of spherical asperity with radius $a_i$</td>
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<td>$A_r$</td>
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<td>$a_i$</td>
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<td>$d_f$</td>
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<tr>
<td>$H$</td>
<td>Hardness</td>
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<td>$H_s$</td>
<td>Hersey number, $\frac{nh}{p}$</td>
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<tr>
<td>$h$</td>
<td>Distance between mean lines of opposing surfaces</td>
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<tr>
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\( h_i \) Indentation depth
\( h_L \) Left ridge height of residual scratch
\( h'_L \) Depth of residual scratch from left
\( h_{\text{max}} \) Maximum depth of wear patch
\( h_R \) Right ridge height of residual scratch
\( h'_R \) Depth of residual scratch from right
\( K \) Wear coefficient
\( k \) Kurtosis
\( L \) Wearing distance
\( L_i \) Characteristic length
\( l \) Distance from substrate to farthest edge of cantilever
\( m \) Average or mean line roughness
\( \dot{m} \) Wear rate
\( m_o \) Original mass
\( N_c \) Number of cycles
\( N_f \) Contact load between fibers using fiber-fiber wear apparatus
\( n \) Strain hardening exponent
\( n_i \) Index of refraction for material \( i \)
\( P \) Applied load
\( P_{\text{lat}} \) Lateral force acting on the cantilever
\( p \) Applied pressure
\( Q \) Volume of worn material
\( Q_i \) Characteristic wear area
\( R_a \) Centerline or arithmetic average roughness
$R_q$  Root mean square (RMS) roughness

$R_{wa/we}^n$ Reaction forces between warp and weft yarns seen at their $n^{th}$ intersection

$r_p$  Pulley radius

$r_s$  Radius of the stationary fiber being worn

$r_{ts}$  Radius at which the torque of the torsion spring is applied

$S_n$  Photodetector setpoint during indentation

$S_t = \frac{(A + B) - (C - D)}{A + B + C + D}$ reading from AFM photodetectors

$S_k$  Skewness

$T$  Nominal torque of torsion spring

$u_0$  Relative speed between indenter and substrate

$u_1$  Relative sliding velocity between indenter and material being displaced

$u_n$  Normal component of surface velocity

$V$  Volume

$V_{worn}$  Wear volume of a patch with dimensions $h_{\text{max}}$ and $w_{\text{max}}$

$W$  Rate of work

$W_{wa}$  Crimp amplitude of the warp yarns

$W_{we}$  Crimp amplitude of the weft yarns

$w$  Width of cantilever

$w_{\text{max}}$  Maximum width of the wear patch

$x$  Horizontal distance

$z$  Axial location along fiber

$\alpha$  Side angles of AFM nanoindentation probe

$\alpha_L$  Slope of left side of residual scratch

$\alpha_R$  Slope of right side of residual scratch

$\gamma$  Angle between translating fiber and vertical
\( \delta_n \) Sensitivity of z-direction piezotube

\( \delta_t \) Sensitivity of the photodetectors

\( \eta \) Viscosity

\( \theta_s \) Output angle of laser reflected off a cantilever

\( \Delta \theta \) Angle of cantilever deflection

\( \Lambda \) Fluid film parameter

\( \mu \) Coefficient of friction

\( \overline{\mu} \) Friction factor

\( \rho \) Density

\( \sigma_0 \) Flow stress

\( \omega \) Rotational speed
CHAPTER 1
INTRODUCTION

1.1 Motivation

There exists a substantial void in the treatment of joint pain associated with localized cartilage damage and wear. Currently, patients presenting with joint pain are asked to endure the pain and are prescribed pain relievers and non-steroidal anti-inflammatory drugs (NSAIDs) to ease discomfort. Other short term pain relief options include rehabilitation, physical therapy, and joint braces [1]. When the pain returns, a surgical procedure is usually performed. There are a number of options including arthroscopic debridement and lavage, marrow stimulation (microfracture), and cartilage transplantation [1–3]. Each of these procedures have associated risks and drawbacks; cartilage transplantation is expensive due to the cost of *in vitro* cultures [4], microfracture does not guarantee repaired cartilage volume or quality [5] and is only effective for relatively small lesions [6], and there are doubts about the effectiveness of arthroscopic debridement and lavage [6, 7]. Even over the counter NSAIDs have side-effects including gastric and duodenal ulceration [1].

These procedures are only temporary and eventually a total joint replacement will be required. During this procedure, the contacting surfaces within the joint are removed and replaced with metal, polymer, or ceramic components, shown in Figs. 1.1 and 1.2.
Figure 1.1. A typical total hip arthroplasty (courtesy DePuy).

Figure 1.2. A typical total knee arthroplasty (courtesy Zimmer, Inc.).
The average age of patients receiving these joint replacements is declining [8,9], due to a number of factors. Cartilage damage and abuse can begin as early as elementary school, when an individual’s articular cartilage is more susceptible to injury as compared to adults [10]. The rise of competitive childhood sports and “extreme sports” over the last 20 years [11,12] has also contributed to the decreasing age of total joint replacement recipients. It is possible for young patients who undergo joint replacement surgery to experience failure of the implant hardware, which only has a lifespan of 15-20 years [13,14]. Even replacements that survive for uncharacteristically long periods of time, some up to 30 years [15], still have the potential to fail within very young patients.

Once joint replacement hardware has failed, the most common solution is a revision joint replacement, which replaces the previous implant. Generally, larger implants are required with larger and more invasive incisions. Although this procedure is not uncommon, it is much more invasive and traumatic than the first joint replacement, with longer recovery times, greater pain, more difficult healing, and an increased risk of adverse outcomes [16,18]. One study conducted in 2005 shows that revision procedures cost 30% more than the primary operation, require 41% more time in the operating room, cause 160% more blood loss, and require 16% longer hospital stays [19]. Similar trends have been found by other studies when comparing primary and revision procedures [20,23]. Revision surgeries are only performed once; if a patient experiences failure of a second joint replacement, confinement to a wheelchair is the most successful way to improve quality of life and decrease pain.

The void in the treatment of joint pain due to cartilage damage exists between the prescription of drugs and the performance of a total joint replacement. Al-
though interim methods exist and are commonly performed, none have proven to be without significant disadvantages. Since total knee replacements are the most commonly performed musculoskeletal related procedure in hospitals since 1998 [24], the potential market for an intermediate procedure is substantial and could greatly improve the quality of life of millions of people. Before patients are required to undergo a full joint replacement surgery, replacing small amounts of damaged cartilage with a synthetic cartilage-like material would permit the patient to live pain-free and would require a less-invasive procedure with shorter and less painful recovery times than a joint replacement. It is feasible that this short-term solution could be performed multiple times with minimal affect on the body. Only small amounts of bone would need to be removed during each procedure and the majority of the natural cartilage would remain intact. Doing so would ensure that future implants and joint replacements would not be compromised. This minimally invasive procedure could only be performed a few times until the amount of remaining natural cartilage becomes insufficient to provide pain-free joint operation. At this time a total joint replacement could be performed with reduced fear of failure within the lifetime of the patient. By delaying the primary joint replacement and eliminating the need for a revision replacement, the amount of pain and discomfort experienced by the patient is drastically reduced while lifestyle and joint usage remain unchanged.

1.2 Proposed Solution

A synthetic plug has been identified as a possible solution to fill the void in the treatment of pain associated with damaged cartilage. A spot of damaged cartilage could be removed and replaced by the proposed plug, shown in Fig. 1.3.
It has been suggested that a three-dimensional woven material would integrate well with natural cartilage due to their similar mechanical properties \[25, 26\]. A design challenge of this novel implant includes mimicking the mechanical and wear properties of cartilage as closely as possible so a seamless integration can be achieved. To do so, an investigation of the function and structure of cartilage is necessary.

Cartilage is a fully hydrated tissue with low stiffness that easily conforms to opposing surfaces. These properties allow it to efficiently transfer load and absorb energy while avoiding damage and maintaining a low coefficient of friction \[27–29\]. Low internal joint friction allows for smooth movement and lower forces required for movement \[30, 31\]. In material science terms, it is truly biphasic; collagen and proteoglycans are the solid phase with water and solutes comprising the fluid which permeates the tissue \[27, 32, 33\].

The orientation of collagen fibers through the thickness of articular cartilage has led to the identification of three layers, seen in Fig. 1.4: the superficial, transition, and deep zones \[34\,39\]. The collagen fiber orientation gradually changes through the thickness of cartilage and provides a variation in mechanical properties necessary for proper function. In the superficial zone, collagen is oriented
parallel to the surface of the cartilage and provides an excellent bearing surface. In the deep zone, collagen fibers are perpendicular to the underlying bone, contain high amounts of hydroxyapatite, and provide solid attachment between the cartilage and the subchondral bone at the tidemark. The transition zone contains collagen in random orientations and serves to gradually change both the structural and mechanical properties between the superficial and deep zones. This gradual change of properties ensures that there are no sharp discontinuities in mechanical properties that could lead to stress concentrations and premature failure.

By using different fiber materials and weave architectures throughout the thickness of a three-dimensional fabric, a gradient in mechanical properties similar to that of cartilage can be achieved. At the base of the woven implant, metal fibers can be used to encourage bone in-growth and to provide a stable foundation similar to the deep zone. Towards the articulating surface, a gradient of fibers will be used to form the transition from metal to polymer, which will comprise the majority of the device. Due to the amount of customization available with a three-
dimensional woven material, the weave patterns and fiber materials will provide a gentle transition of mechanical properties to avoid sharp discontinuities in stiffness. The implant surface must provide low friction and wear, hopefully aided by the porous nature of a woven material that should allow absorption of synovial fluid.

1.3 Thesis Goals

The goal of this project is to investigate the wear characteristics and interactions between individual yarns within a woven material. Micro-motion between fibers will be experienced during each loading cycle and materials will need to be chosen to minimize wear during these micro-motion events. Most of the studies regarding wear of fabrics are limited to composite materials involving a woven pre-form that is infiltrated with an epoxy resin then cured. Limited information is available on the inter-fiber wear experienced within a three-dimensional woven material. The minimization of wear and internal friction is essential for application within a load bearing joint. There are numerous testing procedures that allow wear and the formation of wear debris to be investigated, ranging from simple mechanical testing of the fibers to full-scale prototypes in commercial joint simulators.

According to Fein [41], the confidence in a testing procedure is dependent on the type of test performed and the experience of the researcher. The relationship between test type and the ability to predict performance is shown in Fig. 1.5a. Tests that provide high confidence levels regardless of experience, however, are costly and more time consuming, as depicted in Fig. 1.5b.

In the interest of conserving time and money, it would be beneficial to screen
fibers using methods incorporating material property testing or utilizing a test rig. Fein’s correlations suggest that this could be difficult, especially since the 3D weaving process is a relatively new technology.

This thesis evaluates various screening methods for fibers to be used within the 3D woven device. Testing methods will range from simple mechanical property testing using an atomic force microscope, direct fiber-on-fiber wear using a rig test, to pin-on-disk wear testing performed in an OrthoPOD (AMTI - Watertown, MA). These methods span length scales over seven orders of magnitude, ranging from nanometers to centimeters. Mechanical properties are known to change at very small length scales and it is important to consider not only macro-scale interactions, but also the interplay between surface asperities on the single fiber level.

If a method is found that provides a successful means of fiber screening, future testing times and costs can be significantly reduced. It should be noted that these tests need not provide perfect rankings of all materials. Even methods that
identify fibers that should not be included, as well as those that show potential, are beneficial.
CHAPTER 2

OVERVIEW OF WOVEN MATERIALS

2.1 History

Although woven materials have existed for approximately 30,000 years \cite{42, 43}, their uses and applications are constantly being investigated. Most commonly used for clothing, woven materials have also been used in many other industries. For instance, composite materials utilize weaves as reinforcement in a polymer matrix to provide high strength-to-weight ratios and improved mechanical properties. Simple two-dimensional weaves can be found in sports equipment and personal protective gear, while three-dimensional woven textiles are used in the aerospace industry. Three-dimensional weaves have high specific stiffness, high strength, low weight, good performance and dimensional stability, low thermal expansion, and good corrosion resistance \cite{44}. These outstanding properties, along with the ease of customization, have led biomedical researchers to investigate the use of woven materials in joint and ligament replacements \cite{25, 45, 52}. Overviews of two- and three-dimensional woven materials are given by Mouritz, et al. \cite{53} and Kamiya, et al. \cite{54}. Notable characteristics of woven materials include:

- Near net-shape products can be produced.
- Any material that can be made into a fiber can be woven. Of specific interest are FDA-approved materials, including metals, polymers, and natural fibers.
• The spaces between fibers can be filled with a variety of materials to customize mechanical behavior, interaction between fibers, etc.

• Weave parameters can be varied throughout the volume of the woven material and fibers of different materials can be incorporated to customize mechanical properties, producing functionally graded anisotropic materials.

• The weaving process can be automated to achieve high production rates.

As noted by Mouritz, et al. [53], there are associated drawbacks as well. Three important limitations are:

• Three-dimensional weaves are more expensive to manufacture than two-dimensional ones.

• The physical processes needed to produce three-dimensional weaves are more involved and more difficult than those of two-dimensional ones.

• Modeling the behavior of a three-dimensional woven material is complicated and has not been validated.

2.2 Structure

Woven materials are not a single cohesive unit; instead, they are composed of many individual constituents whose micro-scale interactions determine their macro-scale behavior. Any changes affecting the interplay between fibers will have an effect on the properties and behavior of the material.

All weaves are made of individual strands of material. These strands can be rolled, braided, spun, or formed to produce multifilament yarns, or can be used individually as monofilaments. These mono- or multifilaments are the essential building blocks of all woven materials and can be separated into two categories, warp and weft, which are interlaced to form the most basic woven fabric.

2.2.1 Terminology

There are many terms and measurements commonly used when describing a particular woven structure. A few of the most relevant ones will be explained

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Of particular importance to this research is the “crimp” or “yarn undulation.” These terms refer to the oscillatory formation of the warp and weft yarns after they have been woven together. A sample cross-section of a woven material can be seen in Fig. 2.1, clearly showing the sinusoidal nature of the weft yarns. If a weave is said to have a large amount of crimp, a sinusoidal path with high amplitude is formed and its warp and weft yarns have high $W_{wa}$ and $W_{we}$ values, respectively, which is common for thick yarns. This might also indicate that the fabric is packed very tightly into a dense weave.

When describing the weave pattern used to produce a given fabric, fractional notation is commonly used. The numerator and denominator of the fraction describe the number of warp yarns crossed over and under by a weft yarn, respectively. A “float” within a fabric is a location where the path of two fibers intersect but they do not interlace with one another.
2.2.2 Two-dimensional Patterns

In two dimensions, the most common pattern is the “plain” or “tabby” weave, shown in Fig. 2.2a. The warp and weft fibers follow a simple over-under pattern to form the fabric. This type of weave creates the highest crimp since there are no floats, i.e., fiber crossover occurs at every intersection. The fractional description of a plain weave is 1/1.

Two other common weave patterns are “twill” and “satin,” which can be formed by introducing floats, i.e., by varying the frequency of warp and weft crossover in the fabric. Twill weaves have a characteristic diagonal look as each weft yarn is off-set from the others. Common twill weaves are 2/1, 3/1, and 2/2, but can be produced in a wide variety of patterns. A 3/3 twill pattern is illustrated in Fig. 2.2b. Crimp is usually moderate since the weft yarns do not cross over as frequently as in a plain weave. Satin weaves are defined as architectures in which fibers float over four or more of more of the opposite yarns. An illustration of a 5/1 satin weave is shown in Fig. 2.2c. They have a characteristic shiny appearance and are very smooth since fiber crossover rarely occurs.

2.2.3 Three-dimensional Architectures

To produce a three-dimensional weave, layers are added in the thickness direction of stacked warp and weft yarns and binder yarns are used to hold the layers together. The large number of configurations of weft, warp, and binder yarns provide an essentially unlimited number of options when designing three-dimensional weaves.

Similar to the plain 2D weave, a 3D orthogonal weave is the most simple three-dimensional weave to produce. Seen in Fig. 2.3a, it consists of warp and weft yarns
Figure 2.2. Common two-dimensional woven architectures and their fractional descriptions: (a) plain, 1/1; (b) twill, 3/3; and (c) 5-harness satin, 5/1 (adapted from [57, 58]).

Figure 2.3. Popular three-dimensional woven architectures: (a) orthogonal, (b) layer-interlock, and (c) angle-interlock (adapted from [59]).
bound together by an interlaced binder yarn. Similarly, if the binder yarn does not envelop all layers, a layer-interlock fabric is formed, seen in Fig. 2.3b.

Binder yarns may also traverse the fabric diagonally instead of orthogonally, forming an angle-interlock weave, as illustrated in Fig. 2.3c. As with the orthogonal weaves, the diagonal binder yarns may traverse the entire fabric, or only selected layers.

2.3 Mechanical Testing

The mechanical behavior of a woven material as a whole depends on the mechanical properties of the individual yarns and the interaction between them. The former can be controlled by material selection and the latter is determined by a number of factors, including weave pattern, moisture content, temperature, surface roughness of the yarns, and yarn coatings. Yarn interactions occur over very small surface areas but play a crucial role in mechanical behavior. It has been suggested that a 3D fabric can be uniquely tailored to fit a described set of design requirements with proper choice of material and architectural parameters [60].

2.3.1 Uniaxial Tension

The simplest loading case for a woven material in tension is one in which the applied load is aligned with the warp yarns. In this case, the weft yarns are free to move as the warp yarns deform under the tensile load. As the load is applied to the warp yarns, undulation transfer occurs during which the warp yarns begin to straighten from their naturally crimped position. This causes the weft yarns to further deform around the warp yarns. Since the weft yarns are unloaded, they are free to increase in crimp as they bend around the warp. This
“undulation transfer” is illustrated in Fig. 2.4a; the average displacement of the warp and weft yarns stays the same, hence the yarns stay in complete contact during perfect “undulation transfer.” When the warp yarns have reached their nearly straightened position, yarn extension begins. At this transition, the crimp of the warp yarns has been eliminated and the yarns are aligned with the applied load. Once the warp crimp has been eliminated through undulation transfer, the warp yarns begin to deform under typical tensile conditions without further resistance from the weft yarns.

The load-displacement curve in Fig. 2.4b accounts for both undulation transfer and yarn extension. Under small loads, undulation transfer produces a linear relationship between the load applied to the warp yarns, $P_{wa}$, and displacement. As the warp yarns approach a nominally straight position, they begin the yarn extension phase and elongate linearly with respect to the applied load. During the transition period between these two linear portions, both deformation mechanisms are present, yielding a nonlinear relationship between load and displacement.

Variations of the curve in Fig. 2.4b can be obtained by changing the mechanical properties of the weft yarns, as seen in Fig. 2.5a. Stiffer weft yarns lead to increased...
reaction forces during undulation transfer where the yarns overlap. These higher reaction forces cause a stiffer response of the warp yarns. It can be seen in Fig. 2.5b that the reaction force, $R_{we/wa}$, begins to approach an asymptote as the extension load is increased; this corresponds to complete undulation transfer. Once the warp yarns are perfectly aligned with the applied tensile load, neither the warp nor the weft yarns change crimp. Without further undulation transfer, the internal reaction forces reach their asymptotic value.

2.3.2 Biaxial Tension

During uniaxial tension, the interaction between warp and weft yarns is minimal. Since the weft yarns are not under tension, they are free to deform around the warp yarns, allowing them to become perfectly aligned with the applied tensile force. This is not the case in biaxial tension since the weft yarns are also subject to a load. Both yarn groups attempt to align with their respective applied loads, leading to increased loads and friction between yarns. This tightening of the weave causes the individual yarns to deform from their original cross-sectional geometry.
Figure 2.6. Finite element simulation showing yarn deformation and flattening during biaxial loading of 20 N: (a) unloaded and (b) loaded [61].

to a flatter, more ovalular one, seen in Fig. 2.6. In the example shown, a load of 20 N was applied to both the warp and the weft yarns causing an increase in the spacing of the yarns, a decrease in yarn thickness, and a decrease in cross-sectional area. These changes in yarn geometry led to an increase in volume fraction of the macro-scale material by 18.26% [61].

Recall that under uniaxial loading, the internal reaction forces approached an asymptotic value as the applied load was increased. This is not the case in biaxial loading since both warp and weft fibers are subject to a load. Since neither warp nor weft yarns are able to become fully aligned with the applied tensions, complete undulation transfer is never achieved. Once an equilibrium undulation pattern is reached, internal reaction forces continue to increase linearly with increasing transverse loads, shown in Fig. 2.7. As expected, this increase in internal reaction forces leads to a much stiffer response of the fabric during biaxial tension as compared to uniaxial tension. As the forces between fibers continue to increase, friction and wear become more pronounced.
2.3.3 Pure Shear

Three phases of woven shear response, seen in Fig. 2.8, have been identified and can be described by warp/weft interactions. The relationship between displacement and shear load seems counter intuitive; even at fairly large displacements, little force is required to deform the material. There becomes a point, however, when the material begins to provide resistance to shear deformation and the required load increases very rapidly. In Fig. 2.8, this processes begins around a displacement of 70 mm and becomes pronounced before 90 mm.

During the beginning of shear loading (Phase I), the warp and weft yarns rotate while still maintaining space between them. This simple rotation does not require much force, but as the gap between yarns becomes smaller, Phase II begins and the internal reaction forces begin to increase. As the shear load continues to increase, gaps between the yarns vanish until they cannot be further compressed; this is the point at which wrinkling and Phase III begins. Figure 2.9 illustrates the deformations observed in a textile under shear loading by superimposing displacement vectors on pictures of (a) the fabric and (b-d) individual yarns.
Figure 2.8. Shear load vs. displacement clearly showing the three stages of shear behavior exhibited by fabrics [62].

Figure 2.9. Displacement vectors super-imposed on a fabric during shear testing: (a) overall macro-scale deformation, (b) yarn rotation during Phase I, (c) yarn tightening during Phase II, and (d) yarn compression during Phase III [63].
2.3.4 Abrasion

Initial investigations into the abrasion of fabrics were motivated by physical appearance, not by material longevity concerns. Within the apparel industry, the look and feel of fabric is just as, if not more, important than wear rates and debris production. Initial investigators of fabric abrasion recognized the need to induce uniform wear across the fabric and suggested ways to compare abrasion tests: change in tensile strength, thickness, weight, or porosity [64]. It has been recognized that the wear and abrasion resistance of fabrics is not straightforward and depends on many factors [65]. Given the modeling difficulties, an accepted theoretical approach to woven material wear does not exist and research therefore depends on experimental characterization. In an attempt to standardize testing, an ASTM standard has been developed: ASTM D 4966-10 Standard Test Method for Abrasion Resistance of Textile Fabrics (Martindale Abrasion Tester Method) [66]. This testing apparatus is commonly used to measure pill and visual deterioration of fabrics but was deemed insufficient for the desired uses of the current fabrics. Although it is known that abrasion occurs between fibers and on the fabric surface, quantifying this wear has yet to be performed systematically.
3.1 Friction

The notion of friction on the macro-scale has been well characterized for fairly light loads by the coefficient of friction,

\[ \mu = \frac{F}{P} \]  (3.1)

where \( F \) is the friction force and \( P \) is the applied normal load. This relationship was first proposed by Leonardo da Vinci around 1500 [67], suggested by Guillaume Amontons in 1699 [68], and by Coulomb in 1779 [69] after whom the equation and coefficient are named. The relationship between normal load and friction force described by Eq. (3.1) suffices for most macro-scale systems since the apparent area of contact between surfaces is much less than the real area of contact. This occurs due to surface roughness that causes true contact to occur only between opposing surface peaks. These peaks, also called asperities, play a vital role in all surface interactions including friction, deformation, and lubrication. The macro-scale friction described by Eq. (3.1) is an average over numerous surface asperity interactions. At length scales typical of individual surface asperities, Coulomb friction fails to accurately describe the relationship between friction force and normal load. As the apparent and real areas of contact, \( A \) and \( A_r \) respectively,
approach the same value, the relationship suggested by Eq. (3.1) no longer holds, as illustrated in Fig. 3.1.

When studied at the macro-scale, friction can be viewed as the sum of two contributions: surface adhesion and interference due to deformation or surface roughness. Measuring friction at small scales is beneficial since the contribution of deformation to overall friction can be minimized. Small-scale and low load friction tests allow for investigation of adhesion without the complicating effects of surface deformation. Coefficients of friction found from micro- or nano-scale tests are lower than those from macro-scale tests due to the lack of significant deformation.

In order to quantify a surface it is necessary to define surface measures. These surface measures are used to quantify surfaces and can be correlated to manufacturing process artifacts, lubrication capabilities, and severity of contact stresses. Calculation of these parameters requires discritization of a surface and height values to be measured for each point on the surface, $z_i$. The simplest value to calculate is the mean surface line (in 2D) or plane (in 3D); this value is commonly
TABLE 3.1
NORMALIZED SURFACE PARAMETERS IN TWO DIMENSIONS

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Name</th>
<th>2D Equation</th>
</tr>
</thead>
<tbody>
<tr>
<td>$m$</td>
<td>Average or Mean line</td>
<td>$\frac{1}{n} \sum_{i=0}^{n} z_i$</td>
</tr>
<tr>
<td>$R_a$</td>
<td>Centerline Average Roughness</td>
<td>$\frac{1}{n} \sum_{i=0}^{n}</td>
</tr>
<tr>
<td>$R_q$</td>
<td>Root Mean Square (RMS) roughness</td>
<td>$\left[ \frac{1}{n} \sum_{i=0}^{n} (z_i - m)^2 \right]^{1/2}$</td>
</tr>
<tr>
<td>$S_k$</td>
<td>Skewness</td>
<td>$\frac{1}{n R_q^3} \sum_{i=0}^{n} (z_i - m)^3$</td>
</tr>
<tr>
<td>$k$</td>
<td>Kurtosis</td>
<td>$\frac{1}{n R_q^4} \sum_{i=0}^{n} (z_i - m)^4$</td>
</tr>
</tbody>
</table>

used to normalize all further measurements. A summary of the most common surface parameters is presented in Table 3.1.

3.2 Lubrication

Lubrication can be defined as the use of a substance to reduce friction and/or wear between two opposing surfaces in relative motion. The reduction of friction through the use of a lubricant is achieved by ensuring a certain degree of surface separation and reducing the number of contacting asperities. The degree to which a lubricant separates two surfaces is quantified by the film parameter,

$$\Lambda = \frac{h}{\sqrt{R_{q1}^2 + R_{q2}^2}} \quad (3.2)$$
where \( h \) is the distance between the mean lines of the surfaces and \( R_{q1} \) and \( R_{q2} \) are the root-mean-square roughnesses of the two opposing surfaces. As the film parameter increases, separation between the surfaces increases and asperity contact decreases.

Stribeck [71] experimentally determined a relationship between the coefficient of friction within a journal bearing and the Hersey number, a dimensionless value that can be correlated to film thickness, defined as [72]

\[
H_s = \frac{\eta \omega}{p}
\]

(3.3)

where \( \eta \) is the viscosity of the lubricant, \( \omega \) is the rotational speed of the journal, and \( p \) is the applied pressure. Using the results of Stribeck, Wilson defined three lubrication regimes: boundary, mixed, and hydrodynamic, or full film [73, 74]. These regimes and their corresponding coefficients of friction are displayed in Fig. 3.2 commonly referred to as a Stribeck curve [71]. The following three sections summarize the regimes of lubrication as well as provide a discussion of lubrication within joints.

3.2.1 Full Film Lubrication

For situations where the fluid film parameter is greater than 10, the surfaces are said to be in thick film lubrication where no surface asperity contact occurs and normal load is transferred through a pressurized lubricant film. With no asperity contact, wear of either surface does not occur and friction is very low. As the thickness of the fluid film increases, the friction coefficient increases slightly with some geometries due to limitations in film thicknesses that can be achieved. For film parameters between 3 and 10, a thin film of lubricant separates the mat-
Figure 3.2. The Striebeck curve illustrating the regimes of lubrication for various Hersey numbers and their corresponding coefficients of friction [71, 75].

ing surfaces and prevents asperity contact. Minimal asperity contact sometimes occurs but wear rates are still very low. Both thick and thin film lubrication, known in combination as hydrodynamic lubrication, display very low coefficients of friction and low wear rates. When pressures within the lubricant are high enough to cause elastic deformation of either surface, the system is said to be elastohydrodynamically lubricated.

3.2.2 Mixed and Boundary Lubrication

As the lubricant film shrinks and the film parameter is between 1 and 3, the surfaces are said to be in a mixed lubrication regime in which the surfaces are separated by pockets of pressurized lubricant trapped between direct surface asperity contact. Load transfer is mixed between contacting asperities and pressurized lubricant in the valleys. The direct asperity contact leads to increased friction and higher wear rates than full film lubrication. The transition between full film and mixed lubrication occurs gradually as the surfaces become closer and asperities
begin to contact the opposing surface under higher loads. At film parameters less than 1, the surfaces are in intimate contact with separation due only to the adsorbed films on their surfaces. This situation has been named boundary lubrication by Hardy and Bircumshaw [76]. High friction and high wear rates are inevitable as the surfaces are forced together and asperity contact becomes severe.

3.2.3 Biological Lubrication

Axial forces experienced within a human knee during a normal gait cycle as shown in Fig. 3.3 and range between 0.2 to 2.4 times body weight [77]. It has been proposed that during stance phase compressive stresses force lubricant to exude from the cartilage, providing extra lubrication inside the joint. This extra percolation mechanism could contribute to the low friction and wear observed within natural joints. During swing phase, the compressive stresses are removed and the cartilage is able to absorb the lubricant previously exuded. This cycle is repeated during walking.

Although cartilage is capable of exhibiting a very low coefficient of friction, it has been found that friction increases greatly with increasing loading time [78-81]. Although the exact means by which cartilage exhibits such excellent bearing surface behavior is unknown, many have suggested explanations for these findings, including hydrodynamic lubrication [82], boundary lubrication [83-86], weeping lubrication [80,87,88], boosted lubrication [81,89,90], biphasic lubrication [28,91,93], and micro-elastohydrodynamic lubrication [94]. Although lubrication certainly aides in cartilage tribology, it has also been shown that cartilage performs very well without any synovial fluid [95].
3.3 Wear

Wear is commonly defined as the gradual removal of material from a surface \[96\]. In some cases wear is beneficial, such as in grinding, polishing, cutting operations, graphite pencils, erasers, chalkboards, etc. In most cases, however, wear and its by-products can be detrimental to the operation of a system, leading to increased friction, deterioration of one or both surfaces, and possible component failure. Unlike tensile strength, hardness, and other mechanical properties, the wear resistance of a material is situation dependent. Lubricants, sliding distance, sliding velocity, applied load, surface roughnesses, counter-bearing material, and manufacturing history all play a role in determining the wear resistance of a material. There have been six distinctly different mechanisms of wear identified: adhesive, abrasive, fatigue, impact, chemical, and electrical \[97\]–\[119\]. An in-depth discussion of these mechanisms is provided by Bhushan \[96\].

To-date, numerous machines have been developed for wear testing. Pin-on-disk, reciprocating pin-on-flat, rotating pin-on-cylinder, and crossed cylinders are common testing methods for accelerated wear \[96\]. High cycle wear and fatigue
are most commonly tested using an apparatus such as an OrthoPOD (AMTI - Watertown, MA) by running a desired wear pattern for a number of cycles, usually in the millions. At given intervals, the samples are removed from the machine, dried, weighed, and replaced. Gravimetric measurements allow change in mass during the wearing process to be calculated. Usually, a load-soak sample is utilized to account for moisture uptake. While specimens are being tested, a load-soak sample is submerged in the lubricating fluid and statically loaded. Performing the same gravimetric analysis on this control sample allows for errors due to hygroscopy to be reduced.

Attempts to model the adhesive and abrasive wear mechanisms were made by Archard [120] and are pertinent to the experiments performed in this thesis. The model of adhesive wear assumes a perfectly plastic material with spherical asperities. For each asperity, the cross-sectional area in contact is simply a circle with radius $a_i$,

$$A_i = \pi a_i^2$$  \hspace{1cm} (3.4)

The total contact area is the sum of these individual areas. From the definition of hardness, $H$, applied to each contacting asperity, the normal load, $P_i$, is simply $P_i = HA_i$. The total load is the sum over all contacts; with Eq. 3.4 substituted:

$$P = \sum_i H\pi a_i^2$$  \hspace{1cm} (3.5)

The volume of displaced material is found from the volume of a sphere,

$$V_i = \frac{2}{3} \pi a_i^3$$  \hspace{1cm} (3.6)
and a characteristic wear area can be defined as

\[ Q_i = \frac{V_i}{L_i} = \frac{\pi a_i^2}{3} \]  \hspace{1cm} (3.7)

where \( L_i \) is a characteristic length. Introducing a constant, \( K \), to account for the fact that not every asperity interaction will form a wear particle and summing over all asperities, the Archard wear equation is formed:

\[ V = K \frac{P L}{H} \]  \hspace{1cm} (3.8)

According to this model of adhesive wear, the volume of removed material is proportional to load and sliding distance and inversely proportional to hardness.

In the case of abrasive wear, the model of a plowing conical asperity of radius \( a \) and included angle \( \alpha \) is used and is shown in Fig. 3.4. The material is again assumed to be perfectly plastic and the indenter moves a distance \( L \). The stress under the indenter is equal to the hardness since plastic flow is occurring. The area supporting this load is restricted to the front half of the indenter since the material is perfectly plastic and no recovery occurs after the indenter passes. Via
geometry, it can be shown that

$$\tan \alpha = \frac{a}{h'}$$  \hspace{1cm} (3.9)

where $h'$ is the depth of penetration. From the definition of hardness,

$$P = HA$$

$$= H \frac{\pi a^2}{2}$$

$$= H \frac{\pi (h')^2 \tan^2 \alpha}{2}$$  \hspace{1cm} (3.10)

Solving for $h'$,

$$(h')^2 = \frac{2P}{H \pi \tan^2 \alpha}$$  \hspace{1cm} (3.11)

The cross-section of the plow track is a triangle with height $h'$ and base $2a$. The volume of material removed while sliding a distance of $L$ can be calculated. Substituting Eqs. 3.9 and 3.11 while including a coefficient, $K$, since not every asperity contact will yield a wear event, gives the same relationship as adhesive wear:

$$V = K \frac{PL}{H}$$  \hspace{1cm} (3.12)

It is found that both adhesive and abrasive wear can be described by the same model and are proportional to load and sliding distance while inversely proportional to hardness. This relationship is called the Archard wear equation and is frequently used to quantify wear. Calculation of the wear coefficient is possible by curve-fitting data or through direct measurement under certain testing conditions.
CHAPTER 4
OVERVIEW OF THE ATOMIC FORCE MICROSCOPE

4.1 Introduction

The atomic force microscope (AFM) is a specific example of a scanning probe microscope, or SPM. In general, SPMs rely on the interaction between a probe and sample surface to measure a surface profile, map physical and mechanical properties, or determine surface chemistry.

Binnig et al. developed the first SPM in 1981 [121]. Called the scanning tunneling microscope (STM), it was able to generate a surface profile by using a conducting tip to measure the tunneling current between the tip and sample. A bias voltage was applied to both the tip and sample, thus it was necessary for both to be electrically conductive. A surface profile was generated by monitoring the tunneling current as the surface was scanned. Since the development of the SPM, many researchers have developed more advanced and application-focused machines.

Based on his original STM design, Binnig et al. later developed the atomic force microscope (AFM) which is capable of imaging non-conducting samples [122, 123]. Binnig et al.’s original AFM design was later modified by McClelland et al. [124], Kaneko et al. [126, 128], and Meyer and Amer [129]. Today, commercial AFMs are readily available and come with user-friendly software and easy to use probes.
A simplified diagram of an AFM is given in Fig. 4.1. The driving principle behind the operation of an atomic force microscope is the ability to detect small changes in the deflection of a cantilever. A sharp tip attached to the underside of the cantilever is brought into contact with a surface and the cantilever deflects according to surface topography. A laser beam is focused on the top of the cantilever and reflected onto an array of photodetectors to detect deflection of the cantilever.

A scan is performed by rastering the tip across the sample surface. The movement in the plane of the surface is directed by two piezo transducers, one for each of the \( x \)- and \( y \)- directions. A third piezo is oriented in the \( z \)-direction and allows the AFM head to travel into and out of the plane of the sample surface.
4.2 Basic Capabilities

4.2.1 Imaging

The most common AFM operation is to determine the surface topography of a specimen. There are two settings that can be used on most commercial AFMs to accomplish such measurement: contact mode and tapping mode. Although both of these “modes” have the same theoretical purpose (to profile the surface of a specimen), each mode uses the AFM differently to achieve this goal.

When set in contact mode, a “setpoint” deflection is chosen. This setpoint is defined as \((A + B) - (C + D)\) from the photodetectors shown in Fig. 4.1 and determines the deflection of the cantilever, both of which are held constant during the scan. The tip of the AFM probe is then brought into contact with and moved across the sample surface. When changes in surface topography are encountered, the cantilever deflection is registered via the reflected laser beam on the array of photodetectors. The \(z\)-direction piezo raises or lowers the cantilever to maintain its deflection at the previously defined setpoint. The \(z\)-direction piezo data represents the measured surface profile. Increasing the setpoint improves surface tracking but also increases the force exerted on the sample. The setpoint must be chosen properly to ensure accurate surface tracking is achieved while not damaging the specimen.

When set in tapping mode, the cantilever is resonated. Before imaging can occur, a setpoint amplitude that is less than the resonance amplitude is chosen. The setpoint amplitude is defined as \((A + B) + (C + D)\) from the photodetectors shown in Fig. 4.1 and determines the desired amplitude of the cantilever, both of which are held constant during the scan. The oscillating probe is then lowered to the sample surface until the setpoint amplitude is reached (proximity to the
surface will cause damping of the freely oscillating cantilever, thus lowering its amplitude). As opposed to contact mode, increasing the setpoint during tapping mode decreases the force exerted on the sample. When surface features are encountered, the amplitude of oscillation will change and the $z$-direction piezo will raise or lower the cantilever to hold the oscillation amplitude constant. The $z$-piezo history during a scan generates a surface profile. A typical tapping mode cantilever and substrate are illustrated in Fig. 4.2a.

Although both perform similar tasks, contact and tapping modes each have their advantages and disadvantages. Contact mode is valuable in detecting very small changes in local friction, but while doing so exerts a lateral force on the surface. This lateral force, although small, can be destructive to soft or porous materials. Tapping mode eliminates the lateral force on the sample surface and produces sharp images but does not have friction measuring capabilities. In general, tapping mode is used to image surfaces and contact mode is used for more advanced procedures.

4.2.2 Operation in a Fluid

Although the forces exerted on a sample surface during tapping mode are minimal, very soft materials do not provide the stiffness necessary to resist the tapping motion of the tip. In some cases, surface damage can be incurred by attempting to image soft materials. To overcome this situation, most commercial AFMs can be equipped with a “fluid cell,” seen in Fig. 4.3 which allows a cantilever and substrate to be mounted such that all electrical leads are covered. This allows the AFM to operate within a meniscus of fluid, seen in Fig. 4.3 without damaging the electrical components. Operation in a fluid dampens the resonating frequency
of the cantilever and therefore reduces the forces exerted on the sample surface.

Not only does operation in a fluid allow imaging of soft materials, it also broadens the range of materials able to be used in an AFM to include those of a biological nature. The use of a fluid cell not only benefits imaging, but can also be used more advanced procedures. Hygroscopy can be studied as well as the lubrication effects of various fluids.

4.2.3 Nanolithography

Nanolithography refers to direct control of AFM movement achievable through user-programmed sequences. Written in C and compiled with standard program- ming software, sample operations include:

- $x$- and $y$- piezo (lateral) travel
- $z$- piezo (vertical) travel
Figure 4.3. (a) Bottom and (b) side views of the fluid cell attachment.

Figure 4.4. Illustration of the fluid cell mounted on the AFM. The small meniscus between the sample and fluid cell allows imaging and mechanical testing procedures in a fluid environment.
• Storing setpoint values
• Pausing operation for a specified time
• Ramping peizo voltages to a desired value
• Customizing a GUI

4.3 Mechanical Testing Capabilities

4.3.1 Indenting

When equipped with a special nanoindentation cantilever, illustrated in Fig. 4.2b, a probe can be forced directly into a surface, causing plastic deformation and leaving a permanent indentation. Most nanoindentation probes for use in an AFM consist of a single crystal diamond tip adhesively bonded to the end of a stainless steel cantilever, seen in Fig. 4.5.

Before engaging the surface, the user defines a deflection setpoint, as in contact mode, and the probe is lowered until the interaction between tip and sample cause the cantilever to bend and the deflection setpoint to be reached. Changing the deflection setpoint changes the force exerted on the sample and thus the indentation depth. This procedure is most commonly used for hardness testing and allows shallow indentation depths due to the precision of diamond tips with radii around 40 nm.

Hardness is the resistance of a surface to plastic deformation, and is traditionally defined as the pressure beneath a blunt indenter sufficient to cause plastic flow \[ H = \frac{P_{\text{max}}}{A_c} \] (4.1), or

where \( P_{\text{max}} \) is the maximum load applied during the hardness test and \( A_c \) is the
Figure 4.5. (a) Bottom view and (b) leading edge view of a typical diamond tipped stainless steel cantilever used for mechanical testing in an AFM [131].

Projected area of contact. The normal load applied during testing is given by:

\[ P_{\text{max}} = S_n \delta_n b_n \]  

(4.2)

where \( P_{\text{max}} \) is the maximum normal load applied and usually expressed in nano- or micronewtons, \( S_n \) is the user specified photodetector setpoint in units of volts, \( \delta \) is the sensitivity of the piezotube in units of length per volts, and \( b_n \) is the normal cantilever stiffness, in force per displacement. The area is directly obtained from the tapping mode image of the indentation, and hardness is then calculated using Eq. (4.1).

Indentation experiments can be conducted in an AFM on a small length scale from a few tens of nanometers to a few micrometers of penetration depth. Experimental error can be introduced through inaccurate determination of the applied load and the area of contact during the hardness test. Determination of the applied load is given by Eq. (4.2), but requires accurate knowledge of the cantilever stiffness, which can be determined through beam theory [132–137], fi-
nite element analysis [138–141], static loading [134, 142–149], dynamic response [133, 135, 150, 151], pendulum analysis [134, 142], thermal spectra [152], or experimentally [153]. Reviews of selected methods are presented by Burnham et al. [154], Cumpson et al. [148], and Gibson et al. [155, 156]. All of these approaches have errors and uncertainties associated with them. For example, there is significant adhesive used to affix a diamond tip to a stainless steel cantilever, as seen in Fig. 4.5; the resulting composite beam stiffness is difficult to calculate.

Another difficult aspect of calculating hardness by indentation is determination of the actual contact area between the indenter and material surface. For true hardness calculations, the projected area of contact under the maximum load must be used. Since determination of the area occurs after unloading, this measurement is compromised by any recovery in the material, as well as friction, nonlinear material behavior, residual stresses, etc.

Results from AFM indentations typically display an indentation size effect or ISE. Early observers of this phenomenon include manufacturing researchers investigating grinding processes long before micro/nano testing methods were available. Marshall et al. [157] and Backer et al. [158] commented on the unusually high forces needed during the grinding process, especially for depths of cut less than 25 µm.

To explain these results, a size effect was suggested, similar to that found in other material tensile tests, such as very thin wires [159] and glass fibers [160].

McHargue offers a thorough explanation for the reasons that an indentation size effect exists, including [161]:

- The indentation images in tapping mode may not reflect the projected area of contact in the hardness test because of elastic and viscoelastic recovery of the surface [162, 166]. For example, indentations of PMMA exhibit severe recovery, and the projected triangle will have severely curved sides.
• Indentation shape can be affected by surface residual stresses.

• Depending on the depth of penetration and the shape of the indenter, it is possible for friction between the indenter tip and substrate to affect projected area. Blunt indenters have dead zones beneath them and are less susceptible to friction \[167\]. Since diamond-tipped cantilevers have a finite tip radius, they can act blunt or sharp depending on the depth of penetration achieved. Compensating for friction is a challenging problem in hardness testing \[168–171\].

• Slip-line-field theory provides the fundamental basis for correlating hardness to flow stress \[167\], but slip-line field theory is difficult to apply to strain hardening materials, although it has been attempted \[172\]. Thus, post-yield behavior of the material can make the correlation of hardness experiments to material mechanical properties difficult.

• While normally disregarded, crystal orientation or material anisotropy must be taken into account for small-scale indentations. In addition, it is often unclear how deep a particular grain extends into a surface, so that hardness measurements within a single grain can vary considerably \[173\].

• For materials that exhibit crystalline and non-crystalline structures, commonly observed in many polymers, hardness values will vary depending on which structure is tested.

• Over time, sharp indenters will encounter wear and become dull.

In 1908, Meyer formulated the following relationship to fit the size effect observed in indentation \[174\]:

\[ P = kd^n \]  \hspace{1cm} (4.3)

where \( P \) is the applied load, \( d \) is the diameter of the spherical indenter (or half the diagonal of a Vickers indenter), and \( k \) and \( n \) are constants. McHargue offers a relationship for hardness \[161\]:

\[ H = ch_i^{m-2} \]  \hspace{1cm} (4.4)

where \( h_i \) is the indentation depth and \( c \) and \( m \) are constants.
Bhushan and Koinkar [175] developed a modified AFM to record indentations as shallow as 1 nm which allowed hardness measurements of thin films and materials at the nanometer length scale. In their study, Bhushan and Koinkar found that the hardness of Si(111) increased as the penetration depth and load decreased and attributed this trend to phase transformations beneath the indenter and to films present on the sample surface. Similar results have been obtained by Qian et al. [176] who found that hardness values could vary 10 - 30% between the micro- and nano-scale. The presence of an indentation size effect has a fundamental influence on cutting and plowing mechanics, and is therefore a phenomenon of significant importance to all contacts where wear is a concern.

4.3.2 Scratching

Similar to indentation, scratch testing involves forcing a nanoindentation probe into a sample and translating the tip across the surface. Once a deflection setpoint is specified, the AFM head is lowered until the setpoint is reached, at which point the x- or y- piezos plow the tip through the surface. Scratch testing presents additional experimental challenges compared to indentation testing, but is valuable for the following reasons:

- Plowing directly simulates abrasive friction, a key factor between fibers of the forwarded cartilage replacement.
- Asperity penetration into opposing surfaces is limited to a depth that is approximately one-half of the surface roughness. AFM scratch tests are thus well suited for directly simulating abrasive phenomena in these fibers.
- Contact is restricted to a single asperity and its geometry is well defined.
- The plastic deformation zone in plowing projects ahead of the indenter, as opposed to hardness testing where the plastic deformation zone projects below the indenter. Thus, thinner surface layers can be investigated with scratch testing compared to hardness testing.
The effectiveness of lubricants can be evaluated in a controlled manner under a single asperity sliding contact.

The fundamental modeling of a single plowing asperity was performed by De-Vathire et al. [178] and Azarkhin and Richmond [179–182], modified by Azarkhin et al. [183] and Azarkhin and Devenpeck [184], then further modified by Hector and Schmid [177] to fit the geometry of an AFM indenter, seen in Fig. 4.6. Azarkhin and Richmond [179] expressed the work done in plowing as the sum of energy dissipation due to shear and friction, or

$$W = 2|\vec{u}_1 - \vec{u}_0| \frac{\sigma_0}{\sqrt{3}} (\langle ABD \rangle + \langle BCD \rangle) + 2\bar{\mu} \frac{\sigma_0}{3} |\vec{u}_1| \langle ACD \rangle \quad (4.5)$$

where $W$ is the rate of work, $\bar{\mu}$ is the friction factor, $u_0$ is the relative speed between the indenter and surrounding substrate, $u_1$ is the sliding velocity between the indenter and the material in the tetrahedron ABCD, $\sigma_0$ is the material flow stress, and the angular brackets denote area. Usually, Eq. (4.5) would be minimized to find the solution, but Azarkhin and Richmond state that this technique does not apply to problems with free surfaces due to a lack of kinetic admissibility [185]. Instead, they suggested using a modified upper bound approach using a function.
defined by:
\[ \Phi = \int_S u_n^2 dS \]  
(4.6)

where \( u_n \) is the normal component of surface velocity and \( S \) is the free surface.

Two important results obtained from this model are:

1. Ridge geometry is related to friction factor.
2. Penetration depth is related to material flow stress and is insensitive to friction for sharp indenters such as those in AFMs.

Once the geometry and velocity fields have been obtained through minimization of Eq. (4.6), the plowing force can be obtained through conservation of energy. The input power is the product of material velocity around the indenter, \( u_0 \), and the indenter plowing force, \( F_t \), or

\[ F_t = \frac{W}{u_0} = 2 \frac{|\vec{u} - \vec{u}_0|}{u_0} \frac{\sigma_0}{\sqrt{3}} (\langle ABD \rangle + \langle BCD \rangle) + 2\mu \frac{\sigma_0}{\sqrt{3}} \frac{|\vec{u}|}{u_0} \langle ACD \rangle \]  
(4.7)

The normal force can also be estimated by combining the vertical sliding component of the frictional stresses on surface \( ACD \) and the normal stresses on the projection of \( ACD \), or

\[ F_n = \frac{\sigma_0}{\sqrt{3}} \vec{k} \cdot (\vec{AC} \times \vec{CD}) + 2\mu \frac{\sigma_0}{\sqrt{3}} \left( \vec{k} \cdot \frac{\vec{u}}{|\vec{u}|} \right) \langle ACD \rangle \]  
(4.8)

where \( \vec{k} \) is the unit vector in the z-direction. Equations (4.7) and (4.8) provide estimates of the workpiece strength and friction factor if the forces are known. This is accomplished by writing

\[ \bar{\mu} = \frac{\zeta F_t - \phi F_n}{\psi F_n - \eta F_t} \]  
(4.9)

\[ \sigma_0 = \frac{F_t}{\phi + \bar{\mu} \psi} \]  
(4.10)
where, from Eqs. (4.9) and (4.10), the following expressions have been defined:

\[
\begin{align*}
\zeta &= \frac{1}{\sqrt{3}} \vec{k} \cdot (\hat{AC} \times \hat{CD}) \\
\phi &= \frac{2}{\sqrt{3}} \frac{|\vec{u} - \vec{u}_0|}{u_0} (\langle ABD \rangle + \langle BCD \rangle) \\
\psi &= \frac{2}{\sqrt{3}} \frac{|\vec{u}|}{u_0} \langle ACD \rangle \\
\eta &= \frac{2}{\sqrt{3}} \left( \frac{k}{|\vec{u}|} \right) \langle ACD \rangle
\end{align*}
(4.11)-(4.14)
\]

Other models of an asperity contacting a surface have been proposed by Komvopoulos et al. [186] and Polycarpou and Etsion [187]. These relax the requirement of a sharp tip and allow for conical indenters.

4.3.3 Line Wear Testing

Asperity to surface contact does not generally occur in a single direction for only one cycle, as simulated by scratch testing. Instead, a single asperity is more likely to plow along the same track in both directions during circumstances where repeated cycles are performed. These wear events can be simulated in an AFM by performing reciprocating scratch tests during which the probe is rastered over a line. Naturally, the orientation of the diamond tip is an important variable for these tests. When a sharp edge of the pyramid is aligned with the plowing direction, as indicated in Fig. 4.7a, motion in the reverse direction causes a blunt face to be the leading edge. For more consistent results between the two directions, an orientation as illustrated in Fig. 4.7b is preferred for line testing with the scratch axis perpendicular to the cantilever [188, 189].

After rastering a properly oriented tip over a line for a given number of cycles, tapping mode images of the surface allow for two-fold investigation.
mechanisms can be inferred from the resultant wear track and the morphology of the surrounding area. Second, the volume of worn material can be easily determined and is directly related to wear resistance. The Archard wear equation allows calculation of $K$, the wear coefficient, which can be quantitatively compared between materials. Using the AFM to perform these tests is valuable since they can investigate the wear mechanisms and abrasive wear resistance of materials on the micro scale.

By varying the applied load and number of repetitions performed, one can observe the changing wear mechanisms as surface material is removed. This process has been illustrated by Bhushan and Koinkar [175] using Si(111) samples scratched for 10 cycles under varying loads, shown in Fig. 4.8a. As expected, the resultant scratches show that an increasing load results in an increasing wear depth. Scratch speed has also been investigated; Bhushan and Sundararajan [190] varied the scratch velocity on a Si(100) sample and found that the wear depth is independent of scratch speed. The wear tracks at 1, 10, 25, 50, and 100 µm/s for a normal load of 80 µN can be seen in Fig. 4.8b. It has been proposed that
Figure 4.8. Line wear tracks in (a) Si(111) under increasing load and (b) in Si(100) with increasing velocity \[191\].

The increased frictional effects expected at higher velocities is minimal due to the presence of the large amount of substrate material readily available to dissipate the extra heat produced.
CHAPTER 5

EXPERIMENTAL EFFORTS

Three experimental procedures have been chosen for evaluation as potential screening methods: microscale testing using an AFM, direct fiber-on-fiber wear simulation, and pin-on-disk accelerated wear tests. These tests span numerous length scales and provide increasing experimental complexity. According to Fein’s performance correlations [41], it could be difficult to draw reasonable conclusions from the results of the AFM tests. The results from the fiber-on-fiber rig test should provide more accurate conclusions and the pin-on-disk tests should correlate very well to actual performance. Each testing method is described in this chapter and their results are summarized in the following chapter.

5.1 Micro-scale Testing

It is possible to screen potential fiber candidates for wear resistance by using the Archard wear equation, Eq. (3.8), which relates hardness \( H \), wear volume \( Q \), and loading conditions [120]. Hardness is the only intrinsic material property contained in the equation and thus it follows that hardness can provide a relative ranking of fibers according to abrasive wear resistance. These methods have been successfully used by others to screen thin polymer films [192].
5.1.1 Environment

In order to accurately approximate the wear resistance of a material via mechanical testing, hardness must be evaluated at length scales and in environments similar to those experienced in service. In this case, an in vivo woven cartilage replacement will be exposed to synovial fluid. The effect of synovial fluid on abrasive wear between fibers is unknown but needs to be included in experiments since many polymers of interest are known to be hygroscopic. It is possible that hygroscopy could be beneficial since it has been shown that water can increase the fatigue life of yarns and fabrics [193, 194]. It is well known that the presence of moisture decreases hardness and modulus of most polymers [195–198] but also enhances recovery [199]. Enhanced indentation and scratch recovery would serve to increase the calculated hardness of the material being tested. The interplay between plasticization and the increased material recovery due to moisture is unknown, which is why indentation and scratch testing are performed on dry fibers, fibers within a fluid environment, and hydrated fibers that have soaked in a fluid for an extended amount of time.

For these tests, deionized (DI) water was chosen as the hydrating fluid and fibers were soaked for a minimum of 7 days. This amount of time has been shown to be sufficient for the majority of moisture uptake to occur [200]. Fibers were cleaned with 91% isopropyl alcohol, rinsed with DI water, and dried with compressed air before each test was performed to ensure dirt and oil did not contaminate the contact area. Soaked fibers were cleaned before being immersed in the fluid bath so that cleaning was not needed between removal from the bath and testing, possibly compromising the absorbed moisture within the uppermost surface layers.
A Digital Instruments Dimension 3100 AFM (Veeco - Plainview, NY), seen in Fig. 5.1 was used to perform the experiments in this study. Veeco probes were used for both imaging and mechanical testing and a Veeco fluid cell was used for operations in a fluid environment. Antimony (n)-doped silicon probes, shown in Fig. 5.2 were used for tapping mode images and a stainless steel cantilever with a diamond tip was used for mechanical testing.

5.1.2 Calibration

To allow proper calculation of applied loads during AFM testing, the sensitivity of the piezotube, $\delta$, used in Eq. (4.2) must be determined. To do so, it is common practice to perform an indentation on a very hard material, one that will not plastically deform under the applied load [201]. For these experiments, a sample of quartz was used to make a series of three indentations at varying loads. Average sensitivity values of $163.5\pm1.99$ nm/V and $144.8\pm0.7$ nm/V were found for the
Figure 5.2. Mounted silicon probe for tapping mode use in dry conditions.

dry and fluid operating environments, respectively. The stiffness of the cantilever, \( b_0 \), was found by Veeco to be 720 N/m.

5.1.3 Indentation

Indentations were performed using software provided with the Dimension 3100 AFM. Normal loads ranging from 50 to 1,000 \( \mu \)N were applied at 0.5 Hz with no holding period. After performing the indentations, a silicon probe was used to image the surface in tapping mode. Indentations performed under dry conditions were imaged dry and tests performed in a fluid environment were imaged in a fluid. Hardness was calculated using Eq. (4.1) with \( P_{\text{max}} \) being found from Eq. (4.2) and \( A_C \) measured from the post-indentation surface images. Selected arrays of indentations are shown in Fig. 5.3a and b.

5.1.4 Scratch Testing

Scratch tests were also performed using the Digital Instruments software. Each scratch was made at 0.5 Hz for a length of 15 \( \mu \)m to ensure steady state conditions were reached. Normal loads varied between 25 and 1,000 \( \mu \)N. The lateral deflection
of the cantilever was determined by the use of an oscilloscope connected to a signal access module for the AFM. The lateral deflection of the laser spot, \((A + C) - (B + D)\), was read directly via the oscilloscope and the lateral force acting on the cantilever was found by

\[
P_{\text{lat}} = \frac{b_t \delta_t S_t}{l}
\]  

(5.1)

where \(b_t\) is the torsional stiffness of the cantilever in units of Newton-meters per degree, \(\delta_t\) is the sensitivity of the photodetectors in units of degrees per volt, \(S_t\) is the reading from the oscilloscope in units of volts, and \(l\) is the distance from base of the substrate to the tip of the nanoindentation probe. Subsequent imaging was performed to measure the necessary geometric values for input to the plowing models discussed in Section 4.3.2 [177]. Figure 5.4 contains a sample plow track in PEEK performed with a normal load of 300 \(\mu\)N with the necessary geometric values indicated. Similar to indentation testing, scratches performed on dry samples were imaged under dry conditions and tests performed in a fluid or soaked environment were imaged in DI water. Representative scratches are shown in Fig. 5.3c and d.

5.1.5 Line Wear

Custom nanolithography scripts were written to perform reciprocating line wear tests using the diamond tipped stainless steel cantilever with applied loads between 115 and 1,000 \(\mu\)N. The number of cycles per test ranged between 1 and 500 and velocities between 20 \(\mu\)m/s and 200 \(\mu\)m/s were investigated. One cycle is defined such that the tip travels from its starting position along a 20 \(\mu\)m path perpendicular to the cantilever axis, and returns along the same path.

After performing the tests, tapping mode images were captured of the residual
(a) Depths range from 204 to 101 nm.

(b) Depths range from 105 to 61 nm.

(c) Scratch depths range from 52 to 30 nm.

(d) Scratch depths range from 150 to 110 nm.

Figure 5.3. Representative AFM images of (a,b) indentations and (c,d) scratches performed (material, environment, load): (a) PP, dry, 260 µN, (b) PET, dry, 300 µN, (c) PEEK, fluid, 210 µN, and (d) PET, soaked, 260 µN.
wear tracks. Average surface profiles were taken along the scratches and the depths, heights, and angles of both ridges were measured, corresponding to the variables $h_L, h'_L, \alpha_L, h_R, h'_R,$ and $\alpha_R$ shown in Fig. 5.5, respectively. Large isolated debris and the pileup at the ends of the tracks were not included in the analysis. It is common for only the depth of cut to be measured and used for comparison between tests, but the authors have chosen to use a modified wear “area” instead. Since all scratches were 20 $\mu$m in length and an average cross-section is being taken, comparing wear area is the same as comparing wear volume. It is believed that taking scratch geometry into affect by calculating a wear area will provide more accurate results when compared to scratch depth alone.

To calculate the area of worn material, the geometric approximation superimposed on the measured residual scratch shown at the bottom right of Fig. 5.5 is used. The virgin surface profile is approximated as the dotted line connecting the points where $h'_L$ and $h'_R$ are measured. Using geometry, the following horizontal
Figure 5.5. Worn volume left by reciprocating line wear test in PP after 20 cycles at 350 µN and 20 µm/s and illustration of the geometric approximation for wear area calculation.
distances can be found:

\[ x'_L = \frac{x_L (h_L - h'_L)}{h_L} \quad (5.2) \]
\[ x'_R = \frac{x_R (h_R - h'_R)}{h_R} \quad (5.3) \]
\[ x_L = \frac{h_L}{\tan(\alpha_L)} \quad (5.4) \]
\[ x_R = \frac{h_R}{\tan(\alpha_R)} \quad (5.5) \]

With these values, equations can be formulated for lines approximating the left and right sides of the plow track as well as the original surface profile:

\[ \text{Left} = \frac{h_L (x_L + x'_L)}{x_L} - x \left( \frac{h_L}{x_L} \right) \quad (5.6) \]
\[ \text{Right} = x \left( \frac{h_R}{x_R} \right) - \frac{h_R (x_L + x'_L)}{x_R} \quad (5.7) \]
\[ \text{Top} = x \left( \frac{(h'_R - h'_L)}{x_L + x'_L + x_R + x'_R} \right) + h'_L \quad (5.8) \]

Equations (5.6) and (5.8) intersect at point A and Eqs. (5.7) and (5.8) intersect at point B, as illustrated in Fig. 5.5. These points mark the upper and lower bounds of integration in the x direction for finding the wear volume. It can be shown that the x-coordinates of A and B, respectively, are:

\[ A_x = \frac{2x_L (h_L - h'_L)(2h_L h_R x_L + 2h_L h_R x_R - h_L h'_R x_R - h'_L h_R x_L)}{h_L (2h_L h_R x_L + 2h_L h_R x_R - h_L h'_R x_R - 2h'_L h_R x_L + h_R h'_R x_L)} \quad (5.9) \]
\[ B_x = \frac{h_L h_R}{(2h_L h_R x_L + 2h_L h_R x_R - h_L h'_R x_R - h'_L h_R x_L)(h_R h'_L x_R + 2h_L h_R x_L + 2h_L h_R x_R - 2h_L h'_R x_R - h'_L h_R x_L)} \quad (5.10) \]

These values combined with Eqs. (5.6), (5.7), and (5.8) yield the total wear area
as

\[ A_{\text{worn}} = \int_{A_x}^{B_x} \text{Top} \, dx - \left( \int_{A_x}^{x_L + x'_L} \text{Left} \, dx + \int_{x_L + x'_L}^{B_x} \text{Right} \, dx \right) \] (5.11)

5.2 Fiber-on-fiber Wear Apparatus

Ideally, wear events between weave fibers could be monitored and imaged as the wearing process occurs. This would provide the most accurate assessment of fiber wear within a three-dimensional fabric. Because imaging is difficult within a woven material, the apparatus shown in Fig. 5.6 has been designed to induce controlled wear events, allowing for rapid wear patch location and imaging. One fiber is held stationary while the other is attached to a torsion spring and servo motor (Hitec RCD - Poway, CA), serving to reciprocate a fiber against another. The normal load between fibers can be calculated using the torque in the torsion spring and geometry of the pulleys surrounding the contact area, shown in Fig. 5.6b. The tension in the translating fiber is

\[ F_f = \frac{T}{r_{ts}} \] (5.12)

where \( T \) is the torque in the torsion spring and \( r_{ts} \) is the radius at which the torque is applied to the pulley. The fiber angle, \( \gamma \), can be expressed as

\[ \gamma = \arctan \left( \frac{d_f - r_p}{h_f - h'_f} \right) \] (5.13)

where \( r_p \) is the pulley radius, \( d_f \) and \( h_f \) are the distances from the stationary fiber to the nearest pulleys and base, respectively, and \( h'_f \) is an offset to account for the distance between the base and the bottom of the pulleys. The normal load
between the fibers is

\[ N_f = 2F_f \cos \gamma \]  

(5.14)

Parameters that can be varied in this setup include fiber materials, normal load, sliding distance, and number of cycles. Sliding distances used in the apparatus are significantly longer than would be seen in service and allow for accelerated life testing. The sliding distance has been set to approximately 20 mm, which ensures that a viable portion of the torsion spring is activated to yield smooth and reproducible loading.
5.2.1 Methods and Materials

Two phases of testing were performed; Phase I includes only self-mated monofilament fibers and investigates the effects of speed, load, and number of cycles. Two values of speed and load were chosen as 14 and 63 mm/s, and 0.3 and 1.0 N, respectively. During Phase II, multifilaments are included among the translating fibers and tests were performed using a speed of 63 mm/s and load of 0.3 N. The second phase allowed more materials to be tested without greatly increasing the required number of runs.

During Phase I testing, monofilament poly(ether-ether-ketone) (PEEK) and polyester (PET) were used. Phase II testing included the addition of monofilament titanium (Ti) and nylon, and multifilament fibers of PET, PEEK, and ultra-high molecular weight polyethylene (UHMWPE). Fiber specifications are given in Table 5.2.1. While titanium is not thought to be a viable material for the surface of a cartilage replacement, a metal wire may find use in the substrate weave to provide greater stiffness if desired or to serve as a scaffold for tissue integration. Also, titanium is of interest because of the occasional use of two-dimensional weaves or meshes in-vivo [202–204].

During Phase II, stationary fibers were limited to monofilament PET, PEEK, and nylon to allow for more accurate calculations of wear volume. Although titanium was also available as a monofilament, initial testing confirmed that titanium showed no significant signs of wear when paired with a translating polymer fiber.
<table>
<thead>
<tr>
<th>Material</th>
<th>Density, $\rho$ [g/cm$^3$]</th>
<th>Tensile Strength [MPa]</th>
<th>Surface Free Energy [mN/m]</th>
<th>Hardness, $H$ [MPa]</th>
<th>Fiber Diameter, $d$ [µm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>UHMWPE</td>
<td>0.93</td>
<td>20 - 40</td>
<td>33.5</td>
<td>41</td>
<td>–</td>
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<tr>
<td>Nylon</td>
<td>1.14</td>
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<td>44.3</td>
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<td>300</td>
</tr>
<tr>
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<td>50</td>
<td>44.0</td>
<td>167</td>
<td>250</td>
</tr>
<tr>
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<td>92</td>
<td>42.1</td>
<td>216</td>
<td>150</td>
</tr>
<tr>
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<td>4.50</td>
<td>895</td>
<td>970</td>
<td>100</td>
<td>–</td>
</tr>
</tbody>
</table>
5.2.2 Geometric Model and Statistical Analysis

After performing each experiment, the wear patch on the stationary fiber was examined, with a sample patch shown in Fig. 5.7. Dimensions of the wear patch were measured using optical microscopy since this approach was determined to be the most efficient method for assessing patch size and morphology. Scanning electron microscopy and white light interferometry were considered, but both would have required sputter coating for conductivity and reflectivity purposes. This coating would have interfered with continued testing of the wear patches. The worn volume can be calculated from geometry obtained from patch micrographs using the following model. Two basic assumptions are made; first, the longitudinal profile of the wear patch, shown in Fig. 5.7b, can be approximated as a circular arc. Second, it is assumed that the patch projects uniformly in the $x$-direction. Wear volumes are integrated along the axis of the fiber and compared to an unworn section.

Considering only the top half of a fiber, symmetry is assumed between the two sides, therefore only a quarter of the fiber will be considered. From the measured width of the wear patch, $w_{\text{max}}$, the maximum depth of the patch, $d_{\text{max}}$, is found from the Pythagorean theorem

$$d_{\text{max}} = r_s - \sqrt{r_s^2 - \left(\frac{w_{\text{max}}}{2}\right)^2} \quad (5.15)$$

In the axial direction, the fiber’s thickness variation in the direction of $h_{\text{max}}$ can be obtained as:

$$y(z) = -\sqrt{\frac{4d_{\text{max}}^2 + h_{\text{max}}^2}{64d_{\text{max}}^2}} - z^2 - \frac{d_{\text{max}}}{2} + \frac{h_{\text{max}}^2}{8d_{\text{max}}} + r_s \quad (5.16)$$
Figure 5.7. (a) Top and (b) side views of resulting wear patches after the translating fiber has been removed. The values $w_{\text{max}}$ and $h_{\text{max}}$ are measured directly and $d_{\text{max}}$ is calculated based on fiber radii.

The base of the wear patch in the transverse direction is assumed to be flat; thus an equation for the width of the wear patch is,

$$w(y) = \sqrt{r_s^2 - y(z)^2}$$

where $y$ is defined as the difference between the radius of the stationary fiber and the wear patch depth. The volume of worn material can be found from

$$V_{\text{worn}} = 2 \int_0^{h_{\text{max}}/2} \int_0^w \sqrt{r_s^2 - x^2} - y \, dx \, dz$$

Evaluating the inner integral,

$$V_{\text{worn}} = \frac{1}{2} \int_0^{h_{\text{max}}/2} w(y) \sqrt{r_s^2 - w(y)^2} - \frac{1}{2} r_s^2 \arctan \left[ \frac{w(y) \sqrt{r_s^2 - w(y)^2}}{w(y)^2 - r_s^2} \right] - y w(y) \, dz$$

Equation (5.19) is evaluated numerically.
The volume of removed material can be used in the Archard wear equation, Eq. 3.8 \([120]\), to estimate the wear coefficient and produce a relative ranking of wear resistance. These coefficients will vary depending on material pairings and loading conditions but will allow a quantification of wear events in each situation.

In order to investigate the effects and importance of the various parameters on wear volume and wear coefficient, the commercial software package Design-Expert\textsuperscript{®} (Stat-Ease, Inc. - Minneapolis, MN) was used as a design of experiments (DOE) tool to develop an efficient rubric of tests and to analyze the results via a response surface method. By defining a desired design space and setting upper and lower limits to parameters, a factorial design aids selection of test values. Instead of performing a “full-factorial” experiment, during which values of each parameter are rotated such that every combination is tested, a “fractional-factorial” design is derived in which parameter combinations are chosen to provide sufficient data for response surface generation. Regression analysis of the data provides the response surface and confidence limits. By eliminating the need to test every parameter combination and selectively choosing test values, the number of experiments is greatly reduced without a significant loss of precision. Redundancy is added to further reduce error and to gauge variability in the results.

5.3 Macro-scale Fiber Abrasion Testing \([208]\)

Fiber-on-fiber wear resistance testing has been performed using a rig based on ASTM D 6611-00 \([209]\). Fibers were attached to a hydraulic actuator, guided around a pulley and immersed in a liquid bath. Near the bottom of the container, the fibers were wrapped around another pulley and self-intertwined. The free end was then placed over a third pulley and tied to a weight to provide uniform
tension. De-ionized (DI) water was used as a lubricating and cooling bath and was maintained at 37 ± 2°C to mimic physiological conditions. The hydraulic actuator was set to oscillate at 1 Hz with an amplitude of 50.8 mm.

Wear testing was performed in three phases and each test was run until failure of the fiber due to abrasion occurred or the system reached 5,000 cycles. During Phase I, yarns were tested against themselves at 15% of their tenacity and were twisted together three times. During Phase II, fibers were tested using a constant load of 1.2 N and were intertwined once. During Phase III, different yarns were paired against each other using the same parameters as Phase II. In order to determine appropriate yarn pairings, the fibers were categorized based on their expected location within the final woven material, given in Table 5.2. Fibers from adjacent groups were paired during Phase III, mimicking pairs that could conceivably be woven together in the final device.

### TABLE 5.2

<table>
<thead>
<tr>
<th>In-growth</th>
<th>Transition</th>
<th>Bearing</th>
</tr>
</thead>
<tbody>
<tr>
<td>PEEK</td>
<td>PET</td>
<td>PEEK</td>
</tr>
<tr>
<td>Titanium</td>
<td>PMMA</td>
<td>PET</td>
</tr>
<tr>
<td>PP</td>
<td>PGA</td>
<td>UHMWPE</td>
</tr>
<tr>
<td>PU</td>
<td>UHMWPE</td>
<td></td>
</tr>
</tbody>
</table>

64
TABLE 5.3

FABRICS FOR WEAR TESTING

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Secant Number</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PET-LD</td>
<td>3787</td>
<td>18 “y” ends high, low density in weft</td>
</tr>
<tr>
<td>PET-HD</td>
<td>3786</td>
<td>18 “y” ends high, high density in weft</td>
</tr>
<tr>
<td>PET-S</td>
<td>3796</td>
<td>24 “y” ends high, 2 layers of 5-harness PET satin weave on bearing face</td>
</tr>
<tr>
<td>UH-S</td>
<td>3825</td>
<td>24 “y” ends high, 2 layers of 5-harness UHMWPE satin weave on bearing face</td>
</tr>
</tbody>
</table>

5.4 Pin-on-disk Wear Testing

Samples of three-dimensional woven materials were obtained from Secant Medical® (Perkasie, PA) for pin-on-disk accelerated life testing. Four three-dimensional fabrics have been chosen for wear testing; their descriptions are provided in Table 5.3 along with designations that will be used within this thesis. All fabrics were orthogonal polyester (PET) weaves with single pairs of end stitching. An OrthoPOD (AMTI - Watertown, MA) six station pin-on-disk testing machine is used for the experiments.

5.4.1 Sample Preparation

Circular samples were produced from a large strip of fabric using a punch and mallet. A 35 mm hole punch was used to cut the majority of the sample and a sharp razor blade was used to carefully sever the remaining intact fibers. To prevent fraying, a controlled flame was used to sear the fabric edges.
Fixtures previously used to test two-dimensional woven materials [210] have been modified to accommodate three-dimensional fabrics. The assembly consists of a solid metal puck used as a base with a shallow retaining ring screwed onto the surface, as illustrated in Fig. 5.8. High performance spray adhesive (Loctite - Westlake, OH) was used to adhere the fabric samples to the metal pucks. This method provides enough resistance to prevent sliding and bunching during testing. A stencil was used to ensure the adhesive spray was confined to the circle formed by the retaining ring and the adhesive was allowed to dry to tack before the fabric samples were secured, per the manufacturer’s instructions. Small amounts of overspray were inevitable but were observed to be unchanged at the end of testing and did not interfere with the lubricant or wear of the samples. Fabrics were aligned preferentially so that the direction of lowest friction aligned with the path of the pin. Light pressure was applied and the adhesive was allowed to dry overnight.
5.4.2 Testing Procedures

Cobalt chrome pins with a nominal radius of 12.7 mm were used for wear testing. This is a common material used in orthopedics and may be suitable for direct contact with cartilage [211, 212]. To simulate human gait, a loading and velocity pattern has been modeled from data found in the technical literature [213–219].

With respect to the amount of gait taken by the stance and swing phases, Ayyappa determined each to take 62 and 38 percent of the complete gait cycle, respectively [213]. When testing the frictional properties of a potential cartilage replacement, Covert et al. chose pressures between 0.57 and 1.35 MPa with speeds from 50 to 150 mm/s to mimic walking and running [214].

Although published data varies significantly, the following values were chosen to be representative of a typical gait cycle. A load of 1 MPa was used to simulate stance phase and was reduced to 0.667 MPa during swing phase. A typical sliding velocity of 50 mm/s was chosen during the stance portion of the wear pattern and increased to 81.5 mm/s during the swing portion. Given the size of the pins
and puck samples used in the OrthoPOD, the stance phase of the pattern occurs
over a 20 mm straight line and the swing phase over a 31.4 mm arc, as shown in
Fig. 5.9. Using the velocities specified, a complete cycle takes 0.785 seconds.

Tests were performed in DI water and were stopped periodically for gravimetric
wear measurement using a modified version of ASTM F 2025-06 [220]. Sonicating
cycles including detergent were skipped since testing was performed in DI water
and no chemical cleaning was deemed necessary. The frequency of gravimetric
analysis was lowered as testing progressed.

After preparing the samples and affixing them to the metal pucks, the rubber
O-ring and containment fixture were secured in place. The assembled samples,
shown in Fig. 5.8b, were then filled with 20 mL of DI water, gently agitated to
remove trapped air bubbles from under the fabric surfaces, and allowed to soak
for an hour. Tests were then performed while DI water was replenished as needed.
After the specified number of cycles, the pucks were disassembled and rinsed with
DI water. Each puck was then sonicated in DI water up-side down for 5 minutes,
then rinsed again with DI water. Another sonicating cycle was performed for 15
minutes in DI water with the pucks right-side up. After a final rinse in DI water,
samples were dried with compressed air and placed in a vacuum overnight at 40
°C and 135 Torr. When removed from the oven, samples were allowed to cool
briefly before being photographed and weighed three times in rotation according
to the ASTM standard.

As testing progressed, trends in mass loss, wear rate, and wear coefficient were
observed. Wear rate is defined as

\[ \dot{m} = \frac{m - m_o}{N_c} \]  

(5.20)
where \( m \) is the sample mass, \( m_o \) is the original mass, and \( N_c \) is the cumulative number of cycles. The wear coefficient defined by Archard in Eq. (3.8) is used with a sliding distance per cycle of 51.4 mm and a time averaged load of 9.45 N. The bulk hardness of the individual fiber materials, not of the weave itself, is used for \( H \) and the volume of material removed is calculated using the measured mass loss and known bulk material density.

Load-soak samples were utilized to account for hygroscopy and other effects. While the wear tests were being performed, an extra sample of each material was immersed in DI water and statically loaded. These samples underwent identical cleaning, drying, and weighing procedures as the wear test samples. The change in mass of the load-soak samples was subtracted from the change in mass of the test samples.
CHAPTER 6

RESULTS

6.1 Micro-scale Analysis

6.1.1 Indentation and Scratch Testing

Data from both indentation and scratch tests have been fit to Eq. (4.4) to model the indentation size effect (ISE). A positive ISE was expected, but not all materials exhibited a positive ISE during indentation. A very significant and always positive ISE was found during scratch testing. Indentation and scratch hardness results in each testing environment are shown in Fig. 6.1 for the monofilament fibers. Results for each testing environment are shown in Fig. 6.2.

Statistical analysis has been performed by linear regression of the log-log transformed data. Regression lines were compared using methods described by Zar [221]. Slopes and intercepts were compared using a modified two-tailed t-test and data was concluded to be statistically significant if the calculated p-value was less than 0.05. The results of this analysis are signified by the astricks in Figs. 6.1 and 6.2.

The effects of testing environment are mixed between the PET, PEEK, PP, and nylon fibers. Moisture is known to have a plasticizing effect on polymers that lowers hardness [195–198], but also enhances material recovery [199], which results in an apparent hardness increase. Moisture was found to lower the indentation
Figure 6.1. Indentation and scratch hardness results for (a) PET, (b) PEEK, (c) PP, and (d) nylon monofilaments. Asterisks signify data is statistically different from the two other environments.
Figure 6.2. Hardness results under dry conditions from indentation of (a) bulk, (b) monofilament, and (c) multifilament materials, and results of scratch tests of (d) bulk and (e) monofilament materials. Asterisks signify data is statistically different from all other materials.
TABLE 6.1

RELATIVE RANKING OF MATERIALS DURING DRY-indentATION

<table>
<thead>
<tr>
<th></th>
<th>PEEK</th>
<th>PET</th>
<th>PP</th>
<th>UHMWPE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hardest</td>
<td>Bulk</td>
<td>Mono/multi</td>
<td>Mono</td>
<td>Multi</td>
</tr>
<tr>
<td></td>
<td>Multi</td>
<td>Bulk</td>
<td>Bulk</td>
<td>Bulk</td>
</tr>
<tr>
<td>Softest</td>
<td>Mono</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

hardness of PET and PP, whereas it was found to increase the hardness of PEEK and nylon. The scratch hardness of nylon was unaffected by testing environment whereas moisture was found to increase the apparent hardness of PET and PEEK.

With regard to material form (bulk mono-, or multifilament), it is expected that bulk materials will be softer than the mono- and multi-filament fibers due to polymer chain alignment and increased crystallinity of the fibers during their manufacture. This behavior was observed under both indentation and scratch testing for all materials except PEEK, in which the bulk form was harder than the filaments. These results are summarized in Table 6.1.

6.1.2 Line Wear

After performing the reciprocating line wear tests, the area of removed material was calculated from Eq. [5.19] and used to calculate an Archard wear coefficient. Both wear area and wear coefficient were analyzed by Design-Expert® and a linear model was used to describe each of the response surfaces. Wear area was found
to be dependent on load, material, number of cycles, and only slightly on speed. Wear coefficient was found to be dependent on material, load, and number of cycles.

The relationships between material, load, and number of cycles on wear area can be seen in Fig. 6.3. General trends of increasing wear area with increased cycles and load are observed. For all materials, the wear coefficient was found to drop significantly after the first wear cycle had been performed. For this reason, wear coefficients were calculated at the initial stages of testing and are shown in Fig. 6.4.

Due to the number of significant parameters affecting wear volume, statistical significance and ranking of materials were based upon wear coefficients. Two-tailed t-tests were performed and data exhibiting $p$-values less than 0.05 were deemed significant. Materials on either end of the spectrum, with either very low or very high wear coefficients, have been identified as those requiring further investigation and those that should not be considered, respectively. The wear coefficient of multifilament UHMWPE was found to be statistically different from all other materials except bulk UHMWPE. There was no statistical difference, however, between bulk UHMWPE, multifilament PET, multifilament PEEK, or bulk PET. As for materials exhibiting high wear coefficients, no statistical differences were found between monofilament PET, monofilament PEEK, monofilament nylon, monofilament PP, bulk PP, bulk PEEK, and bulk PET. Also, no differences were found between monofilament PEEK, monofilament nylon, monofilament PP, bulk PP, and bulk PEEK. These results clearly suggest UHMWPE be the primary material for further investigation and that monofilament fibers should be avoided.
Figure 6.3. Wear volumes created during reciprocating line scratch testing.

Figure 6.4. Wear coefficients observed during initial stages of line wear.
6.1.3 Discussion

All hardness values calculated using indentation are more appropriately called “apparent hardnesses” due to recovery of the surface. The materials being used are polymeric in nature and are known to exhibit significant elastic and viscoelastic behavior. When calculating indentation hardness, Eq. (4.1) requires the area of contact, \( A_c \), while the maximum load is applied. Since this measurement is difficult to obtain, it is assumed that there is no recovery during unloading. While this assumption allows accurate hardness measurements of metals and other relatively stiff materials that can be modeled as perfectly plastic, it complicates hardness measurements of polymers and other highly viscoelastic and compliant materials due to the reduced area of the residual indentation. Although these errors are recognized as significant, qualitative hardness comparisons are useful for material evaluation.

There are a few noteworthy trends in the data:

- The relationship between bulk materials during indentation, shown in Fig. 6.2, agrees with generally accepted bulk hardness values.
- Monofilament PET and PP exhibit an uncharacteristic negative ISE. Multifilament PET and UHMWPE exhibit a negative ISE as well.
- Bulk PEEK and UHMWPE exhibit significant ISE during scratch testing and are indistinguishable.
- Monofilament PP exhibits a prominent amount of ISE during scratch testing.
- Results correlate well to the structure of the polymers, illustrated in Fig. 6.5. PEEK and PET have complex structures including one or more benzene rings that inhibit polymer chain motion and result in higher hardness. Those with relatively simple structures, like PP and UHMWPE, can move more easily and are softer. Chemical structure also affects recovery; chains that can move easily exhibit more significant recovery than those with bulky side groups.

Although scratch testing closely mimics the process of abrasive wear [223], sig-
Figure 6.5. Schematic illustrations of the chemical structure of (a) PEEK, (b) PET, (c) nylon, (d) PP, and (e) UHMWPE (adapted from [222]).
significant errors are introduced due to model assumptions and viscoelastic recovery of the residual scratch. Differences in the amount of recovery between materials will greatly affect the calculated hardness values.

Two other concerns arise using the fluid cell, namely the buoyancy of the probe and refraction of the laser through the fluid cell/fluid interface. With regards to probe buoyancy, any forces acting on the probe would serve to lower the load applied to the surface. Calculating the volume of the cantilever and tip, it is found that the buoyant force acting on the probe is approximately 0.01% of the lightest load used during testing. Refraction of the laser beam, however, could contribute more significantly to the differences between hardness values calculated in dry and fluid environments. There is a 12 degree tilt to the cantilever so that the reflected laser can be captured on an array of photodetectors. Under dry conditions, illustrated in Fig. 6.6a, the incident laser reflects off of the cantilever at an angle of 12 degrees. When the AFM is equipped with the fluid cell, a layer of glass is introduced into the path of the laser. This scenario is illustrated in Fig. 6.6b. Using Snell’s Law and the indices of refraction for air, plexiglass, and water, the angle at which the beam exits the top of the fluid cell, $\theta_5$, can be computed. For no cantilever deflection (i.e no load), it is found that there is no difference between dry, fluid cell without water, and fluid cell with water. When a cantilever deflection, $\Delta \theta$, is introduced, however, the exit angle changes according to

$$
\theta_5 = -\sin^{-1} \left\{ \frac{n_3 \sin \left[ 2\Delta \theta - \sin^{-1} \left( \frac{n_1 \sin \theta_1}{n_3} \right) \right]}{n_2} \right\} \quad (6.1)
$$

The relationship between cantilever deflection and exit angle of the laser is shown in Fig. 6.7 for the AFM without the fluid cell, and with the fluid cell with and without water. It can be seen that the glass of the fluid cell has no effect when no
liquid is present. When a fluid is introduced, $\theta_5$ becomes undervalued and results in an apparent decrease of hardness.

Given the wide range of ISE’s encountered, along with the material property variations in each testing environment, an effective ranking of material abrasion resistance cannot be formed. It is clear from the significant scatter, variation between testing methods, and inconclusive results that a more appropriate test of wear resistance is needed. It should be noted that the materials used in this study exhibit very similar mechanical properties and it is not surprising that the differences they show during testing are small.

The results from line wear tests are more promising than simple indentation and scratch tests. Depending on the measures used to rank the materials, either wear area or wear coefficient, slightly different rankings are obtained. When ranking by wear area, the materials are grouped by what form they are in. Multifilaments perform the best, then monofilaments, then the bulk materials. When
wear coefficients are compared, however, multifilaments and bulk materials perform the best, whereas monofilaments perform the poorest.

The superior performance of multifilaments with respect to wear area can be attributed to highly organized polymer chains created by using large drawing ratios during their manufacture. The materials appear to be categorized by decreasing chain alignment; from a high degree in multifilaments, a moderate amount in monofilaments, and a mix of crystalline and amorphous regions in the bulk polymer. The rankings achieved by wear coefficient favor UHMWPE due to its low bulk hardness but surprisingly disfavor the monofilament fibers. It is possible that line wear testing on the micro-scale could serve as a fiber material screening method.

6.2 Fiber-on-fiber Wear Apparatus

6.2.1 Phase I

A two-factor interaction model was deemed significant by Design-Expert® with $R^2 = 0.90$ and was used to describe the response surface for wear volume.
Material, cycles, and load were found to be significant factors while speed was determined to be insignificant. The contour plots in Fig. 6.8 demonstrate the effect of speed, which is noticeable in PET at low cycles but becomes less significant as the number of cycles is increased. For PEEK, it can be seen that speed has little effect throughout the range of cycles tested. Increasing test duration and/or load has the expected result of increasing wear volume, as illustrated in Fig. 6.9a. Under similar testing conditions, PET exhibited the smallest wear volumes.

A linear response surface was used to model wear coefficient. Material and load were the only significant factors and PET exhibited consistently lower wear coefficients than did PEEK, as shown in Fig. 6.9b.

Distinct wear patch and debris morphology were observed from the worn PET and PEEK fibers. Wear patches created between two PET fibers exhibited clearly defined worn volumes with light debris uniformly surrounding them; sample images are shown in Figs. 6.10a and b. Transverse lines across the wear patch were noticeable but not prominent. When PEEK fibers were used, more debris was present and accumulation around the wear patch was not symmetric, as seen in Figs. 6.10c and d. Debris was found attached to one side of the wear patch and the transverse marks within the patch were more significant than with PET.

6.2.2 Phase II

As with Phase I, a two-factor interaction model was used to describe the response surface for wear volume. Both the number of cycles and material of the translating fiber were determined to have significant effect on the wear volume but the stationary fiber material was found to be insignificant.

A linear model was used for the wear coefficient response. The number of
Figure 6.8. Contour lines representing the wear volume response surface for Phase I monofilaments with respect to load and speed.

Figure 6.9. (a) Effect of load and number of cycles on the wear volume of self-mated PET and PEEK monofilament fibers, and (b) wear coefficients calculated from Eq. (3.8).
Figure 6.10. Wear patches for (a-b) self-mated PET fibers and (c-d) self-mated PEEK fibers. Testing variables are 1 N at 63 mm/s for (a) 5 cycles, (b) 1000 cycles, and 0.3 N at (c) 14 mm/s for 500 cycles, and (d) 63 mm/s for 1000 cycles.
cycles and stationary fiber material were found to be insignificant, with translating fiber material having the only significant effect. The insignificance of number of cycles was expected since the Archard wear equation essentially normalizes by sliding distance, $L$, which is directly related to number of cycles in the current experiment. Rearranging the Archard wear equation yields

$$\frac{QH}{P} = LK$$

which is plotted in Fig. 6.11. This sort of normalization allows direct observation of the wear coefficient for each translating material.

As expected, wear patch and debris morphologies for self-mated monofilament tests were similar to those found during Phase I testing. PET produced clean wear patches with little debris and PEEK produced more debris surrounding the patch. When monofilament fibers were paired with one another, debris morphology remained consistent with that of the translating fiber. This finding is in agreement with the statistical insignificance of stationary fiber material with respect to wear
volume and wear coefficient. For example, Figs. 6.12a-c shows stationary PET, PEEK, and nylon fibers after being worn by a translating PET monofilament. All wear patches exhibit characteristics of the PET-PET tests of Phase I. Figures 6.12d-f show the same fibers when worn by a translating PEEK monofilament; wear patch morphologies closely resemble that seen by PEEK-PEEK testing of Phase I. The same is true of the fibers in Figs. 6.12g-i, which have been worn by a translating titanium monofilament. Regardless of the stationary fiber material, the wear volumes created by titanium are significantly larger than those created by a translating polymer fiber.

When multifilaments were used as the translating fiber, wear patch morphology changed drastically. Instead of creating a large depression of removed material, the individual fibrils of the multifilament created tiny wear patches of their own. Optical microscopy revealed that the fiber surface between the patches remained virtually unaltered. This indicates that individual filaments did not move along the axial direction of the stationary fiber during testing, instead their movement was only in the direction of translation. It was noted during testing that the multifilament fiber conformed to the surface of the stationary fiber when loaded. The fibrils arranged themselves such that the geometry of the fiber was more ovular in shape, thus spreading the load over a larger contact area.

Figure 6.13 shows stationary PET, PEEK, and nylon fibers worn by translating PET, PEEK, and UHMWPE multifilaments, respectively. It can be seen that wear debris corresponds to that of the translating fiber. Again, these observations are in agreement with the statistical insignificance of translating fiber material with respect to wear volumes and coefficients.
Figure 6.12. Wear patches from tests involving a (a-c) PET, (d-f) PEEK, and (g-i) titanium monofilaments as the translating fiber during Phase II of testing. Tests were performed using a normal load of 0.3 N and sliding velocity 63 mm/s. Stationary fiber material and number of cycles are (a) PET for 339 cycles, (b) PEEK for 1000 cycles, (c) nylon for 433 cycles, (d) PET for 802 cycles, (e) PEEK for 865 cycles, (f) nylon for 339 cycles, (g) PET for 100 cycles, (h) PEEK for 92 cycles, and (i) nylon for 100 cycles.
Figure 6.13. Wear patches for (a-c) PET, (d-f) PEEK, and (g-i) UHMWPE multifilament as the translating fiber during Phase II of testing. Tests were performed using a normal load of 0.3 N and sliding velocity 63 mm/s. Stationary fiber material and number of cycles are (a) PET for 1000 cycles, (b) PEEK for 100 cycles, (c) nylon for 636 cycles, (d) PET for 644 cycles, (e) PEEK for 303 cycles, (f) nylon for 438 cycles, (g) PET for 906 cycles, (h) PEEK for 559 cycles, and (i) nylon for 267 cycles.
6.2.3 Discussion

Comparing wear coefficients allows a relative ranking of wear resistance to be established. Those fibers with low wear coefficients are attractive for more advanced testing; those with high wear coefficients can quickly be identified as unacceptable candidates. Results from the current study are summarized in Fig. 6.14. It can be seen that multifilament UHMWPE provides the lowest wear coefficient and is thus identified as the most wear resistant of the materials tested. The titanium fiber clearly performed the poorest, causing severe wear of all opposing polymer fibers. Unlike the polymer fibers, the titanium was drawn with a lubricant that became trapped in pockets on the surface of the metal, seen in Fig. 6.15. These pockets formed sharp ridges, a common occurrence for strain-hardened metals during squeeze film operations like drawing [224], which increased the roughness of the titanium fiber. Typical RMS roughness of the titanium fiber surface is 125 nm, compared to the polymer fibers with RMS values of 30 nm (PP), 35 nm (UHMWPE), 40 nm (PET), 50 nm (nylon), and 80 nm (PEEK). The sharp titanium asperities combined with the high material hardness resulted in rapid polymer wear. Roughness of the polymer samples has much less effect since roughness is most likely decreased within the first few cycles of testing [225].

When contact between two polymer samples is considered, adhesion is likely to play an important role. It is hypothesized that adhesion and the highly plastic nature of the samples contributed to the debris formation and buildup. Based on the insignificance of stationary fiber material on debris morphology, it is likely that significant amounts of debris transferred between fibers. This could explain why debris found near the wear patch was dependent only on translating fiber material. Any debris from the stationary fiber was transferred to the translating
one and carried away from the contact area.

The possibility of material compaction and densification inside the wear patch has also been investigated. It is possible that the material assumed to be worn away is instead plastically deformed and compacted below the wear patch. If this were the case, the material at the bottom of the patch would have different mechanical properties. Indentation tests were performed inside a wear patch and on the fiber outside the patch. There was no statistical difference found between the two groups of indentations. It is assumed that the material lost in the wear patch was through a combination of abrasion and adhesion.

The results of these tests are in agreement with previous experiments that identified PET and UHMWPE as materials deserving further testing [208]. The previous tests also indicated a mono-and multifilament pair would provide better abrasion resistance than two monofilaments. Although this result was observed in the previous experiments, it was unclear why this occurred. The visual results of the current study reveal that a multifilament will deform via fibril rearrangement even under very light loads. Each fibril then acts as a translating fiber and creates its own wear patch. These patches are much smaller and support significantly less load. In effect, the volume of removed material for a multifilament is distributed more along the axis of the stationary fiber whereas the volume for a monofilament is concentrated directly under the contact area. Although the wear volume might not significantly decrease, or in fact might increase, the time to failure will be extended since the volume of lost material is distributed along the axis of the fiber.
Figure 6.14. Wear coefficients for each of the translating fibers in Phase II of testing and their respective rankings. Single unpaired asterisks denote statistical differences when compared to all other materials.

<table>
<thead>
<tr>
<th>Rank</th>
<th>Material</th>
<th>K ($\times10^4$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>UHMWPE_{multi}</td>
<td>0.00139</td>
</tr>
<tr>
<td>2</td>
<td>PET_{mono}</td>
<td>0.214</td>
</tr>
<tr>
<td>3</td>
<td>PEEK_{multi}</td>
<td>0.588</td>
</tr>
<tr>
<td>4</td>
<td>PET_{multi}</td>
<td>0.628</td>
</tr>
<tr>
<td>5</td>
<td>PEEK_{mono}</td>
<td>1.42</td>
</tr>
<tr>
<td>6</td>
<td>Titanium_{mono}</td>
<td>47.8</td>
</tr>
</tbody>
</table>

Figure 6.15. (a) Surface topography and (b) cross-section of the titanium monofilament.
### TABLE 6.2

**MATERIALS LASTING 5,000 CYCLES ON THE MACRO-SCALE TEST RIG**

<table>
<thead>
<tr>
<th>Phase I</th>
<th>Phase II</th>
<th>Phase III</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\text{PET}_{\text{mono}}$</td>
<td>$\text{PEEK}_{\text{multi}}$</td>
<td>$\text{UHMWPE}<em>{\text{multi}} - \text{PET}</em>{\text{mono}}$</td>
</tr>
<tr>
<td>$\text{PEEK}_{\text{multi}}$</td>
<td>$\text{PGA}<em>{\text{multi}} - \text{PET}</em>{\text{mono}}$</td>
<td></td>
</tr>
<tr>
<td>$\text{UHMWPE}_{\text{multi}}$</td>
<td>$\text{UHMWPE}<em>{\text{multi}} - \text{PET}</em>{\text{mono}}$</td>
<td></td>
</tr>
<tr>
<td>$\text{PEEK}_{\text{mono}}$</td>
<td>$\text{UHMWPE}<em>{\text{multi}} - \text{PEEK}</em>{\text{mono}}$</td>
<td></td>
</tr>
<tr>
<td>$\text{PU}_{\text{mono}}$</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### 6.3 Macro-scale Fiber Wear Test

Fibers that remained unbroken for the 5,000 cycle testing period are summarized in Table 6.2. Statistically significant differences between the abrasion resistance of mono- and multifilaments were unable to be determined due to differences in loading conditions required to induce failures. Polymers paired with titanium did not perform well due to surface roughness reasons discussed previously. The results do suggest, however, that the most wear resistant pair of materials will be a mono- and multifilament couple.

Three types of UHMWPE were tested: UG 396, SGX 396, and SGX 198. T-tests were performed on the Phase I results of these three materials to determine the effect of filament count and total denier. No significant difference was found between either of the two variables, therefore all UHMWPE results have been combined in this analysis.
6.4 Pin-on-disk Testing

Gravimetric results for the four fabrics tested are shown in Fig. 6.16a. Corresponding wear coefficients of each sample were calculated from Eq. (3.8) at the time of failure and are given in Fig. 6.16b. All samples with a PET bearing face failed before 1 million cycles whereas the UH-S fabric lasted for over 3 million cycles and showed no signs of continued wear or failure. Testing was stopped at 3 million cycles as the UH-S fabric clearly demonstrated superior wear characteristics.

In addition to collecting gravimetric wear data, observation of the sample surface provides insight to the methods of debris production and sample failure. During initial stages of testing, only a light depression was formed on the surface of the samples, seen in Fig. 6.17. After about 50,000 cycles, microscopic fiber failure was observed on the PET-HD and PET-LD fabrics, whereas fiber flattening was observed on the PET-S and UH-S samples. After this initial stage of fibril
rearrangement and flattening on the satin fabrics, very little change is noticed on the surface of UH-S. As wear continues on the other samples, however, severe fiber damage and failure develops, eventually removing the top layers of fabric. This progressive failure and removal of individual layers of fabric is shown in Fig. 6.18.

Large tufts of worn material were observed at the end of the 20 mm stance phase on the PET-HD samples, seen in Fig. 6.19. Damage continued to remove layers of fabric until the metal pin directly contacted the metal puck substrate. At this point, the test was stopped. The evolution of damage for the PET-LD and PET-HD samples is shown in Fig. 6.20 and the PET-S and UH-S in Fig. 6.21.

It was also noticed during testing that the pins used against PET samples were noticeably pitted. Roughness measurements were taken before and after testing using a white light interferometer (Zygo - Middlefield, CT) and are shown in Fig. 6.22. RMS roughness for the pins against the PET samples increased from 25.3 ± 17.1 to 240.3 ± 83.3 µm whereas the RMS for pins against UHMWPE only increased from 26.5 ± 3.5 to 92.5 ± 38.9 µm. On average, roughness increased by
Figure 6.18. Micrographs of wear path illustrating the gradual removal of fabric layers from PET-HD.
Figure 6.19. Optical images of “tufts” formed at the leading edge of the wear patch on the PET-HD samples.

a factor of about 10 for the PET fabrics while only by a factor of about 3.5 for the UH-S samples.

6.4.1 Discussion

The results from pin-on-disk testing clearly identify the UH-S fabric as the most wear resistant. Early failure occurred for all samples with PET bearing faces, regardless of bearing face weave architecture or the density of fibers in the picks direction. The presence of a run-in period was observed for the UH-S sample, lasting approximately 200,000 cycles, after which wear became essentially negligible. Wear of the PET faced samples remained steady until failure. No evidence of a run-in period was observed as the samples exhibited a constant wear rate throughout the test.

The success of UH-S appears to be two-fold, due to both the material of the fibers and to the satin architecture of the bearing face. UHMWPE is the most
Figure 6.20. Evolution of wear in the (a-c) PET-HD and (d-f) PET-LD fabric samples.
Figure 6.21. Evolution of wear in the (a-c) PET-S and (d-f) UH-S fabric samples.
### TABLE 6.3

RESULTS FROM ACCELERATED LIFE TESTING OF UHMWPE

<table>
<thead>
<tr>
<th>Reference</th>
<th>Wear Pattern</th>
<th>Material*</th>
<th>Wear Rate [mm$^3$/km]</th>
</tr>
</thead>
<tbody>
<tr>
<td>196</td>
<td>5x10 mm rectangle</td>
<td>Non-XL</td>
<td>0.056</td>
</tr>
<tr>
<td></td>
<td></td>
<td>XL</td>
<td>0.017</td>
</tr>
<tr>
<td></td>
<td>Unidirectional</td>
<td>Non-XL</td>
<td>0.0037</td>
</tr>
<tr>
<td></td>
<td></td>
<td>XL</td>
<td>0.0195</td>
</tr>
<tr>
<td>226</td>
<td>Bidirectional</td>
<td>Non-XL</td>
<td>0.0904</td>
</tr>
<tr>
<td></td>
<td></td>
<td>XL</td>
<td>0.036</td>
</tr>
<tr>
<td></td>
<td>Multidirectional</td>
<td>Non-XL</td>
<td>0.102</td>
</tr>
<tr>
<td></td>
<td></td>
<td>XL</td>
<td>0.030</td>
</tr>
<tr>
<td></td>
<td>Unidirectional POD</td>
<td>Non-XL</td>
<td>0.0029</td>
</tr>
<tr>
<td></td>
<td></td>
<td>XL</td>
<td>0.0044</td>
</tr>
<tr>
<td></td>
<td></td>
<td>XPLE</td>
<td>0.005</td>
</tr>
<tr>
<td>227</td>
<td>Multidirectional POD</td>
<td>Non-XL</td>
<td>0.0485</td>
</tr>
<tr>
<td></td>
<td></td>
<td>XL</td>
<td>0.069</td>
</tr>
<tr>
<td></td>
<td></td>
<td>XPLE</td>
<td>0.0148</td>
</tr>
<tr>
<td>228, 229</td>
<td>5x10 mm rectangle</td>
<td>Unaged</td>
<td>0.035 - 0.0709</td>
</tr>
<tr>
<td>230</td>
<td>5x10 mm rectangle</td>
<td>Non-XL</td>
<td>0.3546</td>
</tr>
<tr>
<td></td>
<td></td>
<td>XL</td>
<td>Undetectable</td>
</tr>
<tr>
<td>231</td>
<td>5x10 mm rectangle</td>
<td>Unaged XL</td>
<td>0.039 - 0.202</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Unaged Non-XL</td>
<td>0.39 - 0.475</td>
</tr>
<tr>
<td>Current work</td>
<td></td>
<td>PET-HD</td>
<td>3.452</td>
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<tr>
<td></td>
<td></td>
<td>PET-LD</td>
<td>1.939</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PET-S</td>
<td>1.791</td>
</tr>
<tr>
<td></td>
<td></td>
<td>UH-S</td>
<td>0.117</td>
</tr>
</tbody>
</table>

*XL = cross-linked, XPLE = irradiated and cross-linked
common orthopedic polymer used today and is known for its excellent wear resistance. A review of the literature has been performed and wear rates from various UHMWPE tests are provided in Table 6.3. It can be seen that the wear rate of UH-S in the current study is comparable to data from non-crosslinked UHMWPE found in the technical literature.

It is possible that the superior wear resistance of UHMWPE is due in part to material transfer, which is particularly important when polymer on metal wear is considered [232]. It has been noted that there is a large difference between material transfer and the development of a “transfer film” [233]. A thick, lumpy film has been found to be detrimental to wear due to the effective increase in counterface roughness [232]. A thin polymer film, on the other hand, can greatly reduce wear by reducing surface roughness and creating a polymer-on-polymer sliding system. The type and morphology of transfer depends on load, speed, environment, topography, and counterface material [233].

A film is developed during repetitive loading cycles that cause the surface of
the polymer to shear and attach to the opposing surface \[234\]. This attachment is due to both surface roughness and surface energy, with transfer occurring from the material with low cohesive energy to one with a higher cohesive energy \[234, 237\].

With regard to the two materials being tested in the current study, PET has been found to form a patchy film with sections of the undersurface remaining bare \[238, 239\]. PET has also been found to exhibit increased wear under water lubricated conditions during pin-on-disk testing due to plasticization of the polymer \[240\]. UHMWPE has the ability to form thin, surface covering films depending on the testing parameters \[233, 234\]. Although no UHMWPE film is formed when tested in serum, significant UHMWPE transfer was found on a stainless steel surface under DI water and saline solution \[232, 231\]. It is possible that a beneficial polymer transfer film was created by the UHMWPE samples whereas a detrimental lumpy transfer layer was formed by PET, thus causing the significant difference in wear performance.

The success of a satin weave is in sharp contrast to many other studies in the technical literature. Vishwanath finds that plain 2D weaves outperform satin ones during standard pin-on-disk testing \[242\]. It was found beneficial to maximize the number of fiber crossovers in order to hold the fabric together. When crimp is low and crossover occurs less often, as in a twill or satin weave pattern, the fabric is easier to pull apart. These results are applicable to multidirectional wear but for wear in which one direction is heavily favored, like the one used in the current study and in orthopedic applications, the benefits of low crimp in the sliding direction could outweigh the benefits of maximized fiber crossover.

When subjected to fretting wear, it has been found that plain 2D weaves outperformed twill and satin ones \[57\]. This was attributed to the ability of the
Figure 6.23. Micrograph of the edge of the wear path on a satin fabric after 400,000 cycles. Left of center are yarns outside the wear patch and right of center are yarns that have been flattened. No layers of fabric have been removed but distinct yarn flattening is noticeable.

plain weave to trap and retain debris from pulverized fibers. The relative high crimp of the plain weave increased the pore spaces within the fabric, allowing large amounts of debris to be collected.

The results of the current study suggest that lower crimp and fewer warp/weft crossovers are beneficial for sliding in which one direction is heavily favored. With true multidirectional sliding, this observation may not be true. Oscillating fiber cross-over every few fibers also allows the weft fibers more freedom to flatten and rearrange under pressure. This fiber flattening has been directly observed, as shown in Fig. 6.23 and is considered the most beneficial aspect of satin weaves.

The reasons for the increase in roughness of the metal pins during the current studies are unclear. As early as 1955, Hastings observed large amounts of scoring on resected metal samples from joint replacements [243]. Bone cement particles have been found in resected acetabular cups and were believed to be introduced
during implantation or gradually as the device aged [244, 245]. In addition to bone cement particles, silica particles in the UHMWPE [246], and particles of bone [247] have also been cited for scratching metal components. A more recent study of cemented and uncemented implants found scratched metal in both situations [246]. It was then hypothesized that dislodged metal or silica imperfections in UHMWPE were the cause of the scratches. Similar results have been found by others [248] however the exact cause of the scratching is not clear. Scratched metal components have also been observed in commercial joint simulators [249, 251] and accelerated wear tests like the ones contained in this thesis [247, 252, 253].
CHAPTER 7

CONCLUSIONS

The previous studies have demonstrated the practical difficulty of abrasion resistance screening of fibers being investigated for use in a synthetic cartilage replacement. Conclusions from each experimental method are discussed here.

7.1 Micro-scale

Indentation and scratch testing performed using an atomic force microscope have shown inconsistent results between testing methods and materials within each test. Although there are many errors, both fundamental and introduced via the hardware, it was hypothesized that relative wear resistance rankings could be determined. Scatter within the data was significant and the method failed to differentiate between the fibers and environments used during testing.

It is important to recognize that all materials used during nanohardness testing have very similar material properties. Slight differences in mechanical properties are caused by variations in polymer chemistry and structure, however all materials are thermoplastic polymers. It is thus not surprising that differences between material behavior was unable to be determined. The greatest source of error was due to the significant elastic and viscoelastic recovery of the materials. For indentation testing, the calculation of hardness depends directly on the measured area of
the indentation while the load is applied. Since this measurement is unachievable using an AFM, the residual area is used and is subject to recovery upon removal of the normal load. During scratch testing, the model used to calculate hardness is based on slip-line field theory, a method that is well suited for perfectly plastic materials. The scratching process more closely mimics asperity contact during abrasion but is also known to exhibit high degrees of elastic recovery [254, 255]. Given the more pronounced recovery of scratches, especially at small depths of cut, an over exaggerated ISE is measured. The method of scratch testing mimics abrasion well between material pairs with a severe hardness mismatch, but does not truly mimic abrasion between polymers.

Complicating the indentation and scratch procedures is the non-negligible surface roughness of the fibers. Although considered fairly smooth on the macro-scale, the scales of surface roughness and testing depths are very similar. It is possible to embed fibers in a curing polymer, section the puck in a plane along the axis of the fiber, then polish the section to a more acceptable roughness. This procedure is well accepted, however the current studies were meant to focus on the fiber surface. The surfaces of the fibers will be in contact within the final woven product and any unnecessary modification prior to testing was avoided.

When comparing the results of indentation and scratch testing to the more advanced screening methods, it is interesting to note that the materials showing promise, namely PET and UHMWPE, exhibit a negative ISE during indentation testing. This finding is intriguing, however it is clear that a more efficient and accurate screening method for polymer fibers is needed.

Although standard indentation and scratch testing are deemed an unreliable method for screening fibers, reciprocating scratch tests provided more promising
results. As discussed previously, rankings according to wear area favor multifilament fibers, presumably due to their high degree of polymer chain alignment. The results according to wear coefficient, however, are aligned well with the results of more advanced testing, citing UHMWPE, PET, and PEEK as favorable materials.

An interesting phenomenon occurred during line wear testing of PP that should be noted. It was not uncommon for the material to exhibit pilling on the ridges in the form of noticeable clumps. Figure 7.1 shows two examples of such behavior. This type of behavior occurred frequently but was not present on all tests performed on PP. The source of this phenomena is unknown but has previously been attributed to stick-slip or a built up edge [177, 256].

It is suggested that further testing include line wear performed at higher normal loads and larger depths of cut. Although wear tracks produced by normal loads of 120 µN were measurable, increasing the applied loads will minimize errors due to surface roughness. Calculation of wear area and wear coefficient is encouraged and including more materials will aide in the ultimate evaluation of this potentially
beneficial screening method.

7.2 Macro-scale

The macro-scale test for abrasion provided little data about abrasion resistance between yarns [208]. There was no statistical difference found between the various UHMWPE fibers used, but the test was inconclusive when mono- and multi-filament yarns were paired. Titanium and PP were clearly identified as poor candidates for the final implant, however the test failed to identify fibers for potential inclusion. The test rig was difficult to set up and failures not due to abrasion were common. Tests ran for an hour and a half, which was not long enough to differentiate between some materials. For such a time consuming test with minimal results, a more effective screening method is desired.

7.3 Fiber-on-Fiber Simulation

A custom test rig was manufactured and successfully isolated wear between two fibers. It was determined that wear volumes were independent of velocity and stationary fiber material, and a relative ranking of materials was formed based on wear coefficient. UHMWPE and PET were identified as potential candidates and titanium clearly performed poorly. This screening method has significant potential for identifying candidate materials for inclusion in the proposed device.

For a more in-depth investigation into the wear properties of the fibers, a few modifications to the current method are proposed. First, a more accurate calculation for wear volume is needed when multifilaments are used as the translating fiber. Currently the micro-wear patches are counted and the wear volume calculation developed for monofilaments is used on each one. A more accurate method to
determine wear volume under these circumstances would be a great improvement to the system.

A major shortcoming of the current method is the lack of consideration for wear that occurs on the translating fiber. Although this has the potential to be significant, the focus of the current studies was only the stationary fiber. It is possible that inclusion of the translating fiber wear could alter the relative material rankings. It is strongly suggested that this be an improvement made for more advanced testing.

It would be interesting to investigate the effects of wearing a translating fiber against a stationary multifilament. This situation would require the development of a new wear volume calculation method but would make test method more robust.

Although the rig isolates wear very easily between fibers, it does not perfectly mimic the motions that would be experienced in vivo. This is acceptable for a screening method meant for quick and efficient evaluation of fibers, but it could be beneficial to develop a rig that simulates realistic motion between two fibers in which both are moving simultaneously. A new method for calculating wear volume would be needed since wear would no longer be isolated to a specific point on the stationary fiber.

7.4 Pin-on-disk

The use of three-dimensional woven materials in the accelerated pin-on-disk tests have proven to be a very effective method for determining the feasibility of a given material under loaded bearing applications. In a perfect study, weaves with the same architecture would be produced using different materials, thus allowing
the previous methods to be verified or dismissed. Unfortunately, this testing method was not able to fully verify the previous potential screening methods since the availability of three-dimensional woven materials was limited.

In spite of this, however, many significant findings were made simply by using the four materials described previously. The performance of UH-S surpassed all expectations and showed no signs of failure even after 3 million cycles. From these results, UHMWPE has clearly demonstrated superior wear resistance over PET when paired against a metal counterface. Regardless of weave architecture, it appears from the current results that PET is the inferior material.

In order to fully confirm the finding that the success of UH-S was due solely to the inclusion of UHMWPE on the bearing face, a 1/1 plain UHMWPE weave should be tested. If such a sample were to perform as well as UH-S, then material choice would be identified as the prominent factor in wear resistance. If the sample were to fail around 1 million cycles, it would be clear that a proper combination of material and weave architecture need to be chosen for wear resistant fabrics.

Testing fabrics with a combination of monofilament and multifilament fibers could also be performed. Although these types of fabrics would not be expected to perform well, comparing the difference in wear progression could lead to valuable insight and might open doors to new applications for three-dimensional woven materials.
CHAPTER 8

FUTURE WORK

It should be noted that AFM testing most appropriately mimics wear experienced at the surface of the fabric when paired with a metal counterface, whereas fiber-on-fiber testing isolates wear expected to occur within the fabric itself. It is impossible to separate the contributions of bearing surface wear and inter-fiber wear during pin-on-disk tests.

In order to tailor the materials and architecture of the final device, the author suggests that future work be focused on modifying and improving the fiber wear apparatus while performing additional pin-on-disk tests if additional samples become available. Fiber-fiber tests will allow optimization of materials within the weave whereas pin-on-disk tests will allow bearing face material and architecture choices to be explored.

As described in the Introduction, the ultimate goal for the device is to interface with cartilage with minimal damage. A challenge with the overall project will be ensuring native cartilage is not destroyed if these plugs are implanted within a joint. The pin-on-disk tests contained in this work and advanced commercial joint simulators use metal counterfaces opposing the fabric. It should be noted that the studies contained in this thesis focus only on fiber-fiber interaction within the fabric and briefly touch on contact with a metal counterface, however the challenges of interfacing with cartilage are acknowledged.
If it is deemed unreasonable to use a three-dimensional fabric against cartilage for orthopedic applications, the results of the pin-on-disk tests are still encouraging and suggest that woven materials could have applications in other industries. Further research should be performed to investigate their feasibility in bearings and other tribological applications.
REFERENCES


