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Tribological properties of micro/nano-textured surfaces under physiological conditions

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GENERAL DISCUSSION

Due to a decrease in contact area and the ability to store the surrounding fluid inside the structures, micro/nano texturing was expected to reduce dynamic friction between the implant surfaces and human tissue during sliding movement [1-3]. This, however, is not always the case in the low speeds of reciprocating sliding motion in physiological conditions. Increased friction from the textured surfaces can lead to higher risks of irritation and damage (wear) of the surrounding soft tissue in the long term [4,5]. However, a reduction in friction is not the primary aim when applying micro/nano texturing on implant surfaces, instead the aim is to modulate a favorable interaction between the surface and microbial or tissue cells, where increased friction is one of a number of undesirable side effects. Hence, despite the advantageous and promising biological response towards textured surfaces (i.e., *in vitro*), it is critical to study the tribological properties of micro/nano-textured surfaces under a physiological environment (i.e., aqueous environment) before their application onto implant surfaces *in vivo*.

Research in this thesis was carried out to further understand the complex relationships between the tribological properties of the textured hard and soft surfaces (for implants) under physiological conditions. The **key findings** of this thesis as they relate to tribology are shown as the following:

1. If the difference in stiffness is large between the textured and untextured surfaces while sliding, then the texture's edge effect plays a major role in increasing friction.
2. When textures have an order of magnitude difference in size (i.e., nano *versus* micro textures) the larger the texture edge length (being larger for nano textures at the same solid area fraction) the higher the friction. Yet, when texture size is compared within the same order of magnitude (i.e., micro texture), a smaller texture size gives rise to lower friction due to smaller stresses at the texture edges.
3. In general, texturing increases friction under physiological sliding conditions, however the friction can decrease under very specific tribo-conditions, i.e., at a relative high sliding speed, with a surrounding solution with a relative high viscosity, and when a relatively hard probe is used to slide against a relative soft hydrophilic water-containing (i.e., hydrogels) textured substrate.

1. MECHANICAL AND CHEMICAL PROPERTIES OF THE TEXTURED TRIBO-PAIR

In **Table 1**, the details of the material properties of the sliding surfaces (i.e., probe and substrate) used in each chapter of this thesis are listed. Throughout this thesis, we have found that the mechanical properties (i.e., stiffnesses for the tribo-paired objects) and the chemical properties (i.e., hydrophilicity) play an important role in dictating the tribological response.

Table 1. Hydrophobicity with the water contact angles of the tribo-paired objects (i.e., probe and substrate); and the interfacial stiffness (*E'*) between two surfaces of each study in this thesis. $\frac{1}{E'} \equiv \frac{1-\nu_1^2}{E_1} + \frac{1-\nu_2^2}{E_2}$, where E_1, E_2 and ν_1, ν_2 are the Young's moduli and Poisson's ratios for probe (subscript 1) and substrate (subscript 2), respectively.

	Probe			Substrate					Interfacial Stiffness
Chapter No.	Material	Hydrophobicity	Contact Angle, ν°	Material	Hydrophobicity	Contact Angle, ν°		Texturing	*E'*, MPa
Chapter 2	PDMS	hydrophobic	98 – 108 [9]	Si	hydrophilic	20.7 ± 2.7 [10]	with and without	Nano-/ Micro-pores	3.47 [11,12]
Chapter 3	Glass	hydrophilic	4.8 ± 0.4 [13]	pHEMA hydrogel	hydrophilic	~0		Micro-pores	1.60 [14]
Chapter 4	Glass	hydrophilic	4.8 ± 0.4 [13]	PDMS	hydrophobic	98 - 108 [9]		Micro-pores	3.47 [11,14]
Chapter 5	Glass	hydrophilic	4.8 ± 0.4 [13]	pHEMA hydrogel	hydrophilic	~0		Micro-lines	1.60 [14]

*Note: PDMS- polydimethylsiloxane; Si-silicon; pHEMA- poly (2-hydroxyethyl methacrylate); contact angle is 0° means water spread immediately on the surface.

Chapter 3 and **Chapter 4** shared a similar contact mode (i.e., glass probe and *Stiffness*$_{, probe}$ >> *Stiffness*$_{, substrate}$). Hydrophilic hydrogel (i.e., pHEMA, water content 40% by weight) was pore-textured and used as substrate in **Chapter 3**; and in **Chapter 4**, hydrophobic bulk material (i.e., PDMS, no water contained) was used as a substrate. As seen in **Figure 1**, unlike the micropored hydrogel surfaces (which showed a general decrease in friction in **Chapter 3**), an increase in friction was observed with the micropored PDMS when compared to the untextured PDMS in **Chapter 4**. This observation was due to the discontinuity of the liquid film caused by the air-trapped hydrophobic micropores on PDMS. In **chapter 4,** some measurements were performed with textured PDMS being made hydrophilic through mucin and saliva coating (i.e., PGM and RHWS). Yet, the results showed that only RHWS hydrophilic coatings could help to significantly reduce the difference in friction between textured and untextured substrates; friction was still not lower for textured substrates due to the limited liquid retained at the interface by the hydrophilic coatings. Yet in **Chapter 3**, micropored hydrophilic hydrogel had significantly lower COFs than the untextured hydrogel substrate. This strongly indicated that the unbound water inside the hydrogel material and pore textures, which can arrive at the sliding interface due to compression, played a more important role in reducing friction than the bound water on the hydrophilic surface. Still, the results in Chapter 3 and Chapter 4 imply that in an aqueous environment, a hydrophilic surface should be preferred over hydrophobic surface in a tribo-system for a lower friction in general.

In **Chapter 2** and **Chapter 3**, both studies used hydrophilic substrates with and without pore-texturing. Compared to the untextured substrate, micropored textures significantly increased the friction in **Chapter 2** when *Stiffness*$_{, probe}$ << *Stiffness*$_{, substrate}$ (over 10^4 times in difference by Young's modulus); while in **Chapter 3**, similar pore texturing decreased the friction when *Stiffness*$_{, probe}$ >> *Stiffness*$_{, substrate}$ (also over 10^4 times in difference). Such results suggest that the texture's edge effect, which induces the increase in friction by accumulating stresses at texture's edges at the contact interface, plays a more critical role when *Stiffness*$_{, probe}$ << *Stiffness*$_{, substrate}$. Our results suggest, to minimize the risk of high friction from the texture's edge effect, the textured substrate should be softer than the other tribo-paired object [15-17]. Of note, the hydrophilic glass probe used in **Chapter 3** to slide against the hydrophilic hydrogel also helped to decrease friction by allowing more lubricant (i.e., aqueous solutions) into the interface than if the probe was made of hydrophobic material (i.e., PDMS in **Chapter 2**) [18, 19]. Yet, regardless of the texturing, based on the COF values (i.e., COF ~1), the lubrication conditions of all the pHEMA surfaces tested in **Chapter 3** were contained in the boundary lubrication regime (i.e., solid-solid contact, without the formation of a consistent thick liquid film), the hydrophilicity of the glass probe did not play as critical a role as the large stiffness difference of the paired surfaces (i.e., *Stiffness*$_{, probe}$ >> *Stiffness*$_{, substrate}$) in reducing the friction in **Chapter 3**.

Interestingly, in **Chapter 2** and **Chapter 4**, the materials (i.e., bulk material without water content) of the paired objects were flipped, which means that the stiffness and the hydrophilicity were flipped at the same time but with the same interfacial stiffness

(**Table 1**). In **Figure 1**, data from these two chapters show that compared to the untextured substrate, the increase in friction by pore-texturing is more pronounced in **Chapter 2** (when *Stiffness*$_{,\ probe}$ << *Stiffness*$_{,\ substrate}$) than in **Chapter 4** (when *Stiffness*$_{,\ probe}$ >> *Stiffness*$_{,\ substrate}$) under the similar tribological conditions (i.e., sliding speed, applied load and solution viscosity). Such comparison results indicate that the texture's edge effect (caused by the large difference in paired surface stiffnesses) played a more important role for the high frictions than the substrate hydrophilicity in these two studies (as shown in the two sketches of **Figure 2**). Our results suggest that increase in friction from texturing can be more pronounced when *Stiffness*$_{,\ probe}$ << *Stiffness*$_{,\ substrate}$, regardless of the material's hydrophilicity. However, as the Stribeck number increases in **Figure 2** (i.e., with faster sliding speed, more viscous aqueous solution or lower load), the difference in COF between untextured and textured substrates in both studies became closer to each other. This is contributed to the transition of the lubrication regime from the boundary lubrication to the mixed lubrication regime, which implies that the texture's edge effect decreased when more liquid was retained between two paired surfaces.

Therefore, researchers in tissue engineering should be well aware of the possibility of high frictions from texturing on a relatively hard implant surface surrounded by a relatively soft tissue. To minimize the risk of high frictions from texture's edge effect, hydrophilic material with water content should be considered in priority as one of the paired objects; and the material of the textured implant should be softer than its surrounded tissue. However, if the stiffness of the textured implant surface has to be larger than its surround moving/sliding soft tissue, to limit the texture's edge effect the recommended locations of those textured implants should either contain a relatively viscous lubricant (i.e., synovial fluid in knee joints), under a relatively low load, or with a relatively high sliding speed between the two surfaces to achieve a mixed to hydrodynamic lubrication regime.

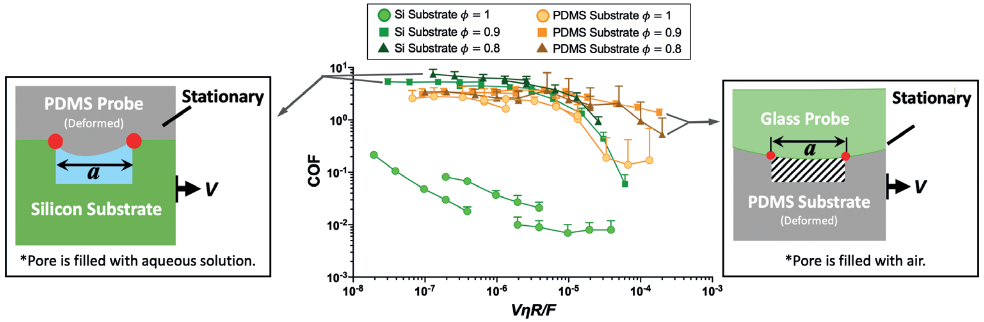

Figure 1. Data comparison in Chapter 2 & Chapter 4: COF presented in the form of Stribeck curves under the similar tribological conditions; and schematic sketches of the side-viewed contact interface of one single pore. Left sketch: **Chapter 2**; right sketch: **Chapter 4**. (Red dots in the sketches represent the texture's edge effect and the size of the red dots reflects the effect intensity.)

2. TEXTURE TYPE OF SHAPE

Other than the pore textures we studied throughout **Chapter 2** to **Chapter 4**, we also studied microlined textures, which have been widely used in biomedical engineering for cell alignment [20-22]. We chose the same material as we used in **Chapter 3**, pHEMA, for our microline texturing study due to its advantage of lowering the friction with the water-contained soft hydrophilic hydrogel. Unlike the micropore textures (**Chapter 3**) which can retain water inside the pores and reduce friction, microline textures did not decrease the friction consistently due to their open ends (open channels, can be also called "microgrooves"), allowing water to easily escape sideways [7,23-25]. In fact, compared to the micropores, the contact interface with the soft hydrogel microlines could be more complicated because of the deformation of the line textures, which could also affect the shape of the microgrooves significantly. The situation in the contact zone with microline texturing was found to be dependent to the applied load and the texture parameters of the lines (i.e., width and depth for both lines and grooves). **Chapter 5** suggests that depending on the texture dimensions, applied load and sliding speed, the channels (microgrooves) can either act as pathways allowing the fluid into the sliding interface or as escape routes for the fluid to leave the contact zone. In order to obtain the best lubricious performance of the microlines when immersed in the aqueous solution, a minimum load or a maximum texture depth should be required to achieve a minimum compression of the line textures on the surface, allowing the solution trapped inside of the grooves to flow immediately upwards to the contact interface, instead of escaping sideways.

The biggest common behavior we found with the soft hydrophilic hydrogel micropores and microlines is that they absorbed water inside of their structures, which they can then in turn supply into the contact zone for lubrication purposes (**Chapter 3** and **5**, and part of **Chapter 4** with protein coating). However, depending on the applied load, as well as the stiffness of the textured substrate and the texture type of shape, the amount of water that can benefit as a lubricant in the interface can be very different from one case to another. For example, if the material of the textured substrate is not adequately soft or the load is not adequately high, the water that is pushed upwards might not be sufficient to lubricate the interface, and a texture type with open ends (i.e., lines) could make the situation worse by expelling water sideways.

As for our fundamental tribological experiments, we could easily change the textured materials and increase the load to achieve a relatively better lubricated interface in the aqueous environment. But in reality, *in vivo*, the properties of the implant materials are dictated by other design considerations, i.e., biocompatibility, fatigue limit etc., and the tissue type will dictate the required mechanical and chemical properties of the surface. In order to minimize the possibility of a high friction induced by the textured surface the only thing which can be controlled are the texture parameters.

3. TEXTURE PARAMETERS

Another important thing we have noticed throughout this thesis is the critical effect of the texture size (i.e., diameter for pores, and width for lines or grooves) on friction, compared to the other texture parameters, such as solid area fraction, texture depth etc. In **Chapter 2** and **3**, the relatively small diameter pores in the micro-scale tend to decrease frictions when compared to the surface textured with a relatively large pore size in diameter; and in **Chapter 5**, surfaces textured with smaller microridges and microgrooves in width, as well, produced lower frictions, under certain conditions. The main reason for the reduction in friction by the texture with a relatively smaller size could be attributed to its relatively steady contact interface; usually, the accumulated stresses at the edges of a relatively smaller texture are relatively smaller (texture's edge effect, **Chapter 2**), which could reduce the chances of the extremely high frictions (i.e., static frictions) at the sliding interface [26-28].

Some studies claimed that if the texture size is very small (i.e., in a few nano meters), the tribological properties of the surface with such textures can almost act the same as the untextured smooth surface, especially when slid against by a large probe (i.e., diameter larger than a few millimeters) [6,29]. But our results show that simply decreasing the texture size may not promise smaller friction. Smaller size could also mean a higher density of the texture edges in the contact area, which can increase the friction significantly when the texture's edge effect is pronounced (nanopores *vs.* micropores in **Chapter 2**). Hence, as the tribological properties of the textured surfaces could be affected by any changes in the tribo-system, the "perfect" texture size with a minimum friction cannot be generalized in a predictable manner.

Hitherto, solid area fraction, which directly reflects the real contact area, was assumed to be an important predictor of the frictional response of a textured surface. The smaller value of the solid area fraction, the less real contact area there is. It was conventionally considered that with less real contact area less friction can be produced. However, early on in our research we realized that this simple assumption may not be true since we found an increase in friction with the textured surfaces, whose solid area fraction was smaller than the solid area fraction of 1 for the untextured surfaces (**Chapter 2**, **4** and **5**). In **Chapter 3**, we also found that at a given diameter for the pore textures, the two textured surfaces with different solid area fractions did not show any significant difference in their tribological properties. Such results forced us to rethink and accept a more critical role played by the texture size over the solid area fraction in tribology.

4. LIMITATIONS OF THIS PRESENT WORK AND OUTLOOK

Our thesis explored the fundamental tribological properties of the textured surfaces by altering their material properties and changing the experimental environment while using the untextured surfaces as control groups. Although the physiological environment was used throughout this thesis, the gap from fundamental research to a real application inside the human body cannot be ignored. More work is necessary to fill this knowledge gap. The next steps could be to move from *in vitro* to *ex vivo* situation, i.e., real pieces of human/animal tissue (such as bone, eye lid, cornea, tongue and cartilage) could be used in tribological experiments to be slid against textured implants' surfaces under the physiological environment. Most of our experiments were performed in phosphate-buffered saline (PBS), but real body fluids should be applied in future studies. In addition, in clinic, many implants are required to be produced with cell culture or cell coculture processes to enhance their biocompatibility before being implanted inside the human body [30-32]. Hence, to come close to the real situation of implant applications, human cells could be cultured or cocultured on the textured surface before subjecting them to tribological tests.

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