Friction and Fracture Characteristics of Engineered Crumb-Rubber Concrete at Microscopic Lengthscale

Ange-Therese Akono a,b,c,* , Jiaxin Chen c, Sakdirat Kaewunruen d,e

a Department of Civil and Environmental Engineering, Northwestern University, 60208, USA
b Department of Mechanical Science and Engineering, University of Illinois at Urbana-Champaign, 61801, USA
c Department of Civil and Environmental Engineering, University of Illinois at Urbana-Champaign, 61801, USA
d Department of Civil Engineering, School of Engineering, University of Birmingham, Birmingham B15 2TT, United Kingdom
e Birmingham Centre for Railway Research and Education, School of Engineering, University of Birmingham, Birmingham B15 2TT, United Kingdom

Abstract

Using small-scale depth-sensing techniques, we shed light on the determinants of friction and hardness in engineered crumb rubber-reinforced concrete with applications into railway sleeper ties. Microscopic scratch tests were carried out to assess the hardness, friction and fracture behavior of concrete specimens reinforced with crumb rubber inclusions. Optical microscopy and scanning electron microscopy are utilized to identify the micro-constituents. The partial replacement of aggregates with crumb rubber particle leads to an increase in the friction coefficient and the fracture toughness and a slight decrease in strength properties. Our research suggests that the crumb rubber particle specific area may play a role in dictating the levels of enhancement in friction coefficient. In addition, improper bonding at the cement/rubber interface is shown to result in poor strength characteristics. Furthermore, crumb rubber particles contribute to a higher durability as evidenced by sustained high values of the friction coefficient even in presence of surface lubrication with water or oil. Overall our study highlights the beneficial role of crumb rubber on the friction and fracture behavior while emphasizing the need for more research into the effect of specific surface area and interface bonding.

Keywords: crumb-rubber concrete, Scratch tests, Hardness, Friction, Fracture Toughness

1. Introduction

Crumb rubber concrete is an alternative way to reuse rubber waste and prevent pollution of the environment [1]. Up to 12 million tons of rubber waste are disposed annually in both the US and Europe [2, 3]. Recycling rubber into advanced construction materials provides a way to alleviate the pressure to landfills. A byproduct of the petroleum engineering industry, tire wastes are estimated at 75 million tons per year in the United States alone [4]. Tire wastes are problematic because (i) they are non-biodegradable, (ii) they require a significant amount of space, (iii) they pose a fire hazard [5], and (iv) they serve as a breeding ground for mosquitoes and larvae. A highly-explored strategy to recycle waste tire consists in embedding crumb rubber in cement mixtures for structural applications such as railway concrete sleepers [6, 7], asphalt pavements [8], or precast concrete [9].

Although previous studies have focused on the strength characteristics of rubber-reinforced concrete [5, 10], the friction characteristics have received little attention. For instance, Liu et al. recorded the mechanical and durability properties of the crumb rubber concrete from the macro level [2]. A negative correlation was observed between the compressive strength and the rubber content [11]. Tuha et al. investigated the mechani-
cal and fracture properties of rubber concrete using quasibrittle fracture mechanics models. They concluded to the existence of an optimal replacement ratio for tire rubber particles to enhance fracture toughness without compromising strength [12]. Ganesan et al. studied the flexural fatigue behavior of self-compacting shredded rubber concrete and showed that a 15 percentage or 20 volume percentage replacement of rubber would significantly improve the distribution of the fatigue life [13]. Ganesan et al. studied the strength and durability characteristics of self-compacting rubberized concrete with or without steel fibers. They found that the addition of steel fibers can compensate the loss of strength due to rubber addition [14]. Nevertheless, in the aforementioned studies, the rheological behavior was not considered. The impact of tire particle/cement matrix bonding was not studied. Finally, the effect of surface treatment on the mechanical performance was not investigated. As friction and wear are important measures of the durability of railway tracks, new studies are needed. To this end, we rely on micro-rheology tests such as scratch testing to gain a fundamental understanding at the micro- and meso-scale.

In order to understand the friction and fracture response, we rely on scratch testing. Other methods such as atomic force microscopy (AFM) [15] and lateral force microscopy (LFM) [16] have been suggested in the past to measure the friction. However, the AFM/LFM techniques present several drawbacks such as tedious force calibration procedure, and unknown probe tip which makes it challenging to gather valuable quantitative information regarding the friction and fracture behavior. Another challenge is the resolution which remains at the nanoscale. In practice, AFM/LFM methods have been used to yield qualitative data regarding the topography and morphology of cementitious materials. For instance, atomic force microscopy (AFM) and lateral force microscopy (LFM) techniques have been employed to investigate the nanostructure and microstructure of cement hydration products [17][18][19]. Herein, we select constant-load and progressive-load scratch testing for its accuracy, reliability and rigor.

Scratch tests consist in pushing a sharp diamond probe across the surface of a weaker material. Scratch tests are frequently used to characterize the friction behavior of metals, polymers, thin films, coatings, and ceramics [20][21][22][23][24]. Very recently, scratch tests have been applied to characterize the tribology of cementitious materials and geomaterials, which exhibit a large degree of heterogeneity [25]. To our knowledge, scratch tests have not yet been applied to crumb rubber-reinforced concrete. A major challenge is the large range of scale between the whole concrete at the meso and macroscopic scale and the micro-constituents at the microscale. Herein, we apply fracture analysis, strength and hardness relationships, and friction analysis to scratch testing in order to understand the tribological behavior of crumb-rubber concrete at different length-scales and under different loading conditions and surface treatment options.

2. Materials and Methods

Four different types of crumb-rubber reinforced concrete were synthesized at the Birmingham Centre for Railway Research and Education at the University of Birmingham. The mix design is summarized in Table 1. Mix 1 is the control material, which consists of cement, water, fine aggregate, and coarse aggregate. Table 2 provides the gradation of the aggregates used in this study. In order to compensate for the potential loss in mechanical resistance due to the addition of crumb-rubber particles, fume silica was introduced in Mix 2–4 at a reason of 10% in weight with respect to the mass of fine aggregates. Mix 2 was reinforced with silica fume whereas both Mix 3 and Mix 4 were reinforced with rubber with a mass fraction of respectively 5% and 10% with respect to the mass of fine
Table 1: Design of crumb-rubber reinforced concrete systems considered in this study.

<table>
<thead>
<tr>
<th>Material</th>
<th>Cement (kg)</th>
<th>Water (kg)</th>
<th>Fine Aggregate (kg)</th>
<th>Coarse Aggregate (kg)</th>
<th>Silica fume (kg)</th>
<th>Rubber (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mix 1</td>
<td>530</td>
<td>233</td>
<td>630</td>
<td>986</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Mix 2</td>
<td>477</td>
<td>233</td>
<td>630</td>
<td>986</td>
<td>53</td>
<td>0</td>
</tr>
<tr>
<td>Mix 3</td>
<td>477</td>
<td>233</td>
<td>599</td>
<td>986</td>
<td>53</td>
<td>32</td>
</tr>
<tr>
<td>Mix 4</td>
<td>477</td>
<td>233</td>
<td>567</td>
<td>986</td>
<td>53</td>
<td>63</td>
</tr>
</tbody>
</table>

Table 2: Aggregate Gradation Table

<table>
<thead>
<tr>
<th>Serial no.</th>
<th>Sieves (mm)</th>
<th>% retained</th>
<th>Cumulative % fine retained</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>20</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>16</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>10</td>
<td>21</td>
<td>21</td>
</tr>
<tr>
<td>4</td>
<td>6.7</td>
<td>67.5</td>
<td>88.5</td>
</tr>
<tr>
<td>5</td>
<td>4.75</td>
<td>9</td>
<td>97.5</td>
</tr>
<tr>
<td>6</td>
<td>Base</td>
<td>2.5</td>
<td>100</td>
</tr>
</tbody>
</table>

Table 2: Aggregate Gradation Table

<table>
<thead>
<tr>
<th>Properties</th>
<th>Specification</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>&gt;90</td>
<td>%</td>
</tr>
<tr>
<td>Retention on 45 μm</td>
<td>&lt;1.5</td>
<td>%</td>
</tr>
<tr>
<td>H₂O (when packed)</td>
<td>&lt;1.0</td>
<td>%</td>
</tr>
<tr>
<td>Bulk Density (U)</td>
<td>200 – 350</td>
<td>kg/m³</td>
</tr>
<tr>
<td>Bulk Density (D)</td>
<td>500 – 700</td>
<td>kg/m³</td>
</tr>
</tbody>
</table>

Table 3: Chemical and Physical Properties of Silica Fume

aggregates. Silica fume, grade 940 was utilized for Mix 2–4 with the chemical and physical properties of silica fume given in Table 3. Two different sizes of crumb rubber were used: 425 μm with a specific gravity of 1.14 ±0.02 for Mix 3, and 75 μm with a specific gravity of 1.14±0.03 for Mix 4. For each design, 5.5-in.×2-in.×1-in. specimen blocks were manufactured. The specimens were subsequently aged for 28 days prior to microscopic examination and testing.

2.1. Material Preparation

In order to ensure accurate measurements, a rigorous specimen preparation procedure was devised so as to yield a low surface roughness relative to the maximum penetration depth [26]. The specimens were machined using a top-table bandsaw and later embedded under vacuum in an epoxy resin. A linear-precision diamond saw was later utilized to yield 5-mm thick cylindrical specimens with rigorously flat top and bottom faces. The resulting specimens were mounted onto metal disks using cyano-acrylate adhesive. The mounted specimens were then ground and polished using a semi-automatic grinder/polisher. Grinding occurred using silicon carbide abrasive discs of different gradations, consecutively 240, 400, 600, 800, and 1200. Afterward, polishing took place using colloidal suspensions of polycrystalline diamond with particle size consecutively 3 μm, and 1 μm. In between each steps of the grinding and polishing phases, the specimens were rinsed in N-Decane using an ultrasonic bath. The quality of the polished
surface was assessed via optical microscopy and surface profilometry. After grinding and polishing, the specimens were stored in a vacuum desiccator at room temperature to prevent water-induced degradation [27].

2.2. Micro-structural Characterization

Scanning electron microscope (SEM) was used to image the polished crumb-rubber cement specimens. A JEOL JSM-6060LV Low Vacuum Scanning Electron Microscope (SEM) was utilized at the Frederick Seitz Materials research Laboratory with an accelerating voltage of 15–20 kV and a working distance of 10 mm. Fig. 1 displays representative SEM images for Mix 3. A matrix-inclusion micro-structure is observed. The matrix phase is hardened cement whereas the inclusions consist of aggregates and rubber particles. The aggregate particles (light grey) are 200–2000 \( \mu \text{m} \) in size. In particular, imperfect bonding is observed between the rubber particles and the surrounding hardened cement matrix.

2.3. Scratch Testing

Constant load scratch tests were applied to characterize the hardness and friction properties. All tests were conducted using a Micro Scratch Testing equipment (MST), that was compliant with the standards ASTM G171, ASTM D7187, and ASTM D7072 [28, 29]. The equipment featured a load resolution of 0.01 mN and a depth resolution of 0.05 nm. The scratch testing unit was integrated with a high-resolution video microscope to allow the precise positioning of the test. As shown in Fig. 2, in our experiments, a spherico-conical diamond stylus was pushed across the surface of the material while applying a constant or linearly increasing vertical force. In all tests, a Rockwell C probe was used, characterized by a tip radius \( R = 200 \mu \text{m} \) and a half-apex angle \( \theta = 60^\circ \). The scratch probe was accurately measured using scanning confocal microscopy. Prior to testing, the specimen surface profile was measured via a surface scan using a contact load of 3 mN. During the test, continuous stiffness measurement was utilized to record the forces and the penetration depth in real time along the scratch path. At the end of each test, a panorama image of the residual top surface was captured. In this study, the temperature was held constant at 72 ± 2 \(^\circ\)F, the testing took place under an acoustic enclosure, and the scratch probe was thoroughly cleaned prior to each tests to prevent debris accumulation.

Table 4 displays the scratch parameters used in this study. A total of 304 scratch tests were performed following eight different protocols. We carried out both meso-scale tests, with a constant load of 15 N, and microscale tests, with a constant load of 1 N. In addition, for progressive-load testing, the vertical force was linearly increased from 0.1 N to 2 N. The meso-scale tests were carried out to assess the effective behavior of each mix (Protocol P1) as well as the influence of surface treatment (protocols P2 and P3). Fracture scratch tests (protocol P4) were performed to evaluate the fracture toughness of each mix design. For protocols P1–P4, the location was selected randomly within a given material specimen, Mix 1–4. In contrast, for protocols P5–P9, in-situ optical microscopy was utilized to select an aggregate, silica, rubber particle or a cement matrix space. Microscale scratch tests (protocols P5 and P6) were performed to measure the contribution of each micro-constituent—aggregate, micro-silica, cement paste, and rubber—to the overall behavior. Finally, we investigated the effect of loading rate and scratching speed on the measured scratch hardness and friction coefficient (protocols P7–9).

3. Theory

3.1. Friction and Hardness

Table 4 defines the mathematical notations employed in this study. The friction and hardness were analyzed following
Figure 1: a) Scanning Electron Microscope images of crumb-rubber concrete cement Mix 4 to identify the micro-constituents. The particle identified are aggregate and silica fume inclusions, in light grey, and rubber, in black.

<table>
<thead>
<tr>
<th>Protocol</th>
<th>$P$</th>
<th>$V$</th>
<th>$X$</th>
<th>Surface Lubricant</th>
<th>System</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>15</td>
<td>6.0</td>
<td>3</td>
<td>None</td>
<td>Mix 1–4</td>
</tr>
<tr>
<td>P2</td>
<td>15</td>
<td>6.0</td>
<td>3</td>
<td>Deionized Water</td>
<td>Mix 1–4</td>
</tr>
<tr>
<td>P3</td>
<td>15</td>
<td>6.0</td>
<td>3</td>
<td>Oil</td>
<td>Mix 1–4</td>
</tr>
<tr>
<td>P4</td>
<td>0.1–2.0</td>
<td>6.0</td>
<td>3</td>
<td>None</td>
<td>Mix 1–4</td>
</tr>
<tr>
<td>P5</td>
<td>1</td>
<td>0.2</td>
<td>0.1</td>
<td>None</td>
<td>Rubb.</td>
</tr>
<tr>
<td>P6</td>
<td>1</td>
<td>2.4</td>
<td>0.2</td>
<td>None</td>
<td>Agg., Cem., Sil.</td>
</tr>
<tr>
<td>P7</td>
<td>0.1</td>
<td>2.4</td>
<td>0.2</td>
<td>None</td>
<td>Agg.</td>
</tr>
<tr>
<td>P8</td>
<td>1</td>
<td>0.4</td>
<td>0.2</td>
<td>None</td>
<td>Agg.</td>
</tr>
<tr>
<td>P9</td>
<td>0.1</td>
<td>0.4</td>
<td>0.2</td>
<td>None</td>
<td>Agg.</td>
</tr>
</tbody>
</table>

Table 4: Scratch protocols for our study. A total of 304 scratch tests was carried out. $P$ is the prescribed vertical load in N. $V$ is the scratch speed in mm/min, $X$ is the scratch length in mm. Agg. = aggregate. Cem. = cement paste. Sil. = silica.
Figure 2: a) Digital photograph of a scratch test. Credits: Ange-Therese Akono, Pooyan Kabir, UIUC, 2016. b) Constant-load scratch test. c) Progressive-load scratch test. d) Scratch probe geometry. \( d \) is the penetration depth, \( F_T \) is the horizontal force, and \( P \) is the vertical force. \( R \) is the probe tip radius, meanwhile \( \theta \) is the half-apex angle and \( w \) is the scratch width.

<table>
<thead>
<tr>
<th>Mathematical symbol</th>
<th>Physical meaning</th>
</tr>
</thead>
<tbody>
<tr>
<td>( A )</td>
<td>horizontally-projected load-bearing contact area</td>
</tr>
<tr>
<td>( \beta )</td>
<td>Weibull shape parameter</td>
</tr>
<tr>
<td>( d )</td>
<td>Penetration depth</td>
</tr>
<tr>
<td>( \Delta \phi )</td>
<td>Increase in porosity due to improper bonding</td>
</tr>
<tr>
<td>( d_t )</td>
<td>scratch probe transition depth</td>
</tr>
<tr>
<td>( F_T )</td>
<td>Scratch horizontal load</td>
</tr>
<tr>
<td>( H )</td>
<td>Hardness</td>
</tr>
<tr>
<td>( I_r )</td>
<td>rubber inter-particle distance</td>
</tr>
<tr>
<td>( K_c )</td>
<td>Fracture toughness</td>
</tr>
<tr>
<td>( \mu )</td>
<td>Apparent friction coefficient</td>
</tr>
<tr>
<td>( \eta )</td>
<td>Weibull scale parameter</td>
</tr>
<tr>
<td>( P )</td>
<td>Scratch vertical load</td>
</tr>
<tr>
<td>( p )</td>
<td>perimeter</td>
</tr>
<tr>
<td>( R )</td>
<td>Probe tip radius</td>
</tr>
<tr>
<td>( r )</td>
<td>size of rubber particles</td>
</tr>
<tr>
<td>( t )</td>
<td>thickness of rubber particles</td>
</tr>
<tr>
<td>( \theta )</td>
<td>Half-apex angle of probe</td>
</tr>
<tr>
<td>( V )</td>
<td>Scratch speed</td>
</tr>
<tr>
<td>( w )</td>
<td>Scratch width</td>
</tr>
<tr>
<td>( X )</td>
<td>Scratch path</td>
</tr>
<tr>
<td>( \phi )</td>
<td>volume content rubber</td>
</tr>
</tbody>
</table>

Table 5: Description of the mathematical symbols used in this study.
ASTM G171-03 [29]. In turn, the scratch hardness provides information regarding the material strength characteristics [30]. The scratch hardness was computed as the ratio of the applied constant vertical force $P$ to the vertically projected contact area:

$$H = \frac{P}{\frac{\pi}{8}w^2} \quad (1)$$

In this study, the vertically projected area, $\frac{\pi}{8}w^2$, is calculated from the scratch width. In turn, the scratch width $w$ is calculated from the measured penetration depth as:

$$w = \begin{cases} 
2\sqrt{R^2 - (R - d)^2} & d \leq d_t \\
2(d - R(1 - \sin \theta)) \tan \theta + 2R \cos \theta & d \geq d_t 
\end{cases} \quad (2)$$

Herein $d$ is the penetration depth that is recorded in real time using high-accuracy sensors, $R$ is the probe tip radius, and $\theta$ is the probe half-apex angle. In particular, $d_t = R(1 - \sin \theta)$ is the scratch probe transition depth from the spherical into the conical domain. Analogously, the friction coefficient $\mu$ is defined as the ratio of the horizontal force $F_T$ to the vertical force $P$:

$$\mu = \frac{F_T}{P} \quad (3)$$

3.2. Fracture Analysis

Nonlinear fracture mechanics was employed to relate the horizontal force $F_T$ to the fracture toughness $K_c$ [31][32][33]:

$$K_c \equiv \frac{F_T}{2pA} \quad (4)$$

Where, $2pA$ is the probe shape function that depends on the geometry of the scratch probe as well as the penetration depth $d$ [32][31]. In our tests, the function $2pA(d)$ was calibrated using a reference materials as described in [32]. $d$ is the penetration depth, which is measured using high-accuracy sensors.

The theoretical model is derived in details in [31][32][33] using the J-integral, the energetic size effect law, and computational fracture mechanics. In particular, the method was validated on polymers, ceramics, and metals [31] and has been applied to characterize the fracture behavior of a wide range of materials including but not limited to cement-polymer composites [34], rocks and cement paste [35], and organic-rich shale [36]. Herein, we apply the scratch fracture method to understand the influence of crumb rubber reinforcement on the fracture behavior.

4. Results

4.1. Individual Test Results

Fig. 3 illustrates the analysis procedure from individual constant-load and progressive-load scratch tests. For instance, consider a single scratch test carried out under a constant vertical load of 15 N. Given the continuous stiffness measurement system, the forces—horizontal $F_T$, and vertical $P$—as well as the depth $d$ are recorded every 3 $\mu$m. Fig. 3 a) displays the continuous evolution of the force and depth profiles along the scratch path $X$. The depth profile $d$ yields the width profile using Eq. (2). In turn, the width can be utilized to compute the hardness along the scratch path using Eq. (1). The force measurements can also be used to compute the friction coefficient $\mu$ as shown. Due to the heterogeneity of the specimen—consisting of hardened cement paste, aggregates, silica fume, and crumb rubber—, large variations occur along the scratch path for both the hardness and the friction. In particular, the maximum penetration depth oscillates between 52 $\mu$m and 95 $\mu$m; the hardness varies between 0.20 and 0.47 GPa, and the friction coefficient varies between 0.06 and 0.56. Thus, each individual constant-load test yields 1,000 independent measurements of the friction coefficient $\mu$ and of the scratch hardness $H$. Similarly, Fig. 3 b) displays the force and depth measurements recorded during a progressive-load test with a maximum vertical force of 2 N. In turn, the penetration depth increases up
Fracture toughness analysis from a single constant-load test with a constant vertical force equal to 15 N. By application of Eq. (4), the fracture toughness can be estimated along the scratch path: $K_c$ oscillates around a mean value of 0.55 MPa $\sqrt{m}$ with a standard deviation of 0.2 MPa $\sqrt{m}$. Thus, each individual progressive-load test yields 1,000 independent measurements of the fracture toughness $K_c$.

4.2. Effect of Scratch Speed and Normal Load

From a method development perspective, it is important to understand the influence of the prescribed normal load and scratch speed on the measured friction coefficient. Similarly, from an application standpoint, different train loads and speed will result in different rates and levels of mechanical loads applied locally. Thus we carried out a set of constant-load scratch tests on the aggregate phase at two different speeds, 400 mm/min and 2400 mm/min, and two different load levels: 0.1 N, and 1 N, following protocols P 6–9 in Table 5. For simplicity, we focused on a single micro-phase: aggregate. Fig. 4 the frequency distribution of the scratch friction coefficient $\mu$ and of the scratch hardness $H$ for both load levels and scratch speeds.

On the one hand, the scratch load alters the shape of the frequency distribution and the median value of the friction coefficient. In particular, a very small increase—only 16%—of the friction coefficient is recorded when the normal load is multiplied by 10. The dependency of the friction coefficient on the applied normal force is similar to AFM-based friction tests carried out by Bhushan et al. on polished silicon, silica, and diamond [37] with nanoscale normal loads. This increase of the friction coefficient with the normal load at the nanoscale is commonly attributed to ploughing. On the other hand, the shape and the and the median value of the frequency distribution is not altered when the scratch speed is increased by 500%.

In the scientific literature, the influence of sliding speed on friction has been linked to the viscoelastic behavior for polymers [38, 39, 40] and rocks [41, 42]. In this case a rate-independent behavior is observed for the friction coefficient showing that for...
the timescales and length-scales of our experiments, and for the aggregate phase, the visco-elastic energy dissipation is negligible compared to friction-induced energy dissipation. Thus, in what follows, we can consider the friction coefficient to be invariant with respect to the loading rate and scratch speed.

4.3. Influence of Individual Micro-constituents

Protocols P4 and P5 were followed to measure the friction and hardness properties of individual micro-constituents: aggregate, silica, hardened cement paste, and rubber. The micro-constituents were selected randomly and tested within specimens from all four mixes Mix 1–4 using optical microscopy. Fig. 5 displays the frequency distribution for both the friction and hardness. For aggregate, silica, and hardened cement paste, the frequency distribution of the friction coefficient exhibits a single peak whereas the frequency distribution of the hardness exhibits several peaks. This difference points to the different nature of hardness—characteristic of strength—with average values of 75 µm and 425 µm explaining the broad range of the resulting friction coefficient. As a result, the hardness is primarily influenced by the composition and the morphology whereas friction is primarily driven by the topology of the surfaces in contact. Thus, the different peaks in the hardness frequency distribution are caused by different types of aggregates, silica inclusions, and different cement hydration products.

We can rank the micro-constituents according to the average friction coefficient: in descending order, rubber, cement paste, silica, and aggregate. Friction is promoted in hardened cement paste due to the presence of nanopores, micropores, along with grains boundaries for the cement hydration products. As for silica, its particulate nature—with a particle size ranging from 75 µm and 425 µm—promotes asperities at the inclusion boundaries. Finally, rubber presents an intrinsically textured surface. This textured surface, coupled with the bimodal particle distribution—with average strength 75 µm and 425 µm—explains the broad range of the resulting friction coefficient.
Figure 5: Friction and Hardness of Individual micro-constituents. (Color online)
In turn, the micro-constituents can be ranked according to their hardness, in ascending order: rubber, cement paste, silica, and aggregate. The reverse order between friction and hardness suggests a compromise between friction and strength, as characterized by the hardness.

5. Discussion

5.1. Synergistic Effects on Friction

Fig. 6 shows the impact of fume silica and crumb-rubber addition on the friction coefficient. The frequency distribution of the friction coefficient is represented for constant-load scratch tests carried out on materials Mix 1–4 following protocol P1. On the one hand, looking at each curve, separately, we observe a synergistic effect. For instance, Mix 1 exhibits values of the friction coefficient greater than 0.5 whereas its basic constituents—hardened cement paste, silica, and aggregate—are characterized by values of the friction coefficient strictly less than 0.3, cf. Fig. 6b, c, d). In other words, due to the high heterogeneity and the large local variations in morphology, the effective friction coefficient is significantly higher than that of each microphase considered individually. On the other hand, we note that the frequency distribution is altered by the presence of silica fume and crumbed-rubber. Finally, each frequency distribution curve presents multiple peaks, which are evidence of a discrete range of friction mechanisms.

Table 6 displays the average values of the friction coefficient for constant-load tests under dry conditions for all four mix designs. The friction coefficient \( \mu \) increases by 1% when fume silica (Mix 2) is added to plain concrete. \( \mu \) increases by 10% when 75-\( \mu \)m crumb-rubber particles are added at a volume fraction of 5% (Mix 3). Finally, \( \mu \) increases by 7% when 425-\( \mu \)m crumb-rubber particles are added at a volume fraction of 10% (Mix 4). Although Mix 3 and Mix 4 represent an improvement in terms of friction coefficient with respect to Mix 1 and Mix 2, the increase in the value of the friction coefficient is not proportional to the volume fraction of crumb rubber. The reason is that friction is a surface phenomenon, as a result, the relevant variable is the specific area \( a \) of rubber particles. Assuming a statistically uniform dispersion, we have \( a \propto \phi_r r^2 \) where \( \phi_r \) is the rubber volume content and \( r \) is the size of rubber particles.

In particular, when comparing Mix 3, and Mix 4, the rubber particles in Mix 3 are in average 5.6 times larger than those in Mix 4; whereas the volume content of Mix 4 is only twice that of Mix 3. Thus, Mix 3 exhibits a specific area which is 15.7 times greater than that of Mix 4, which explains why the increase in friction coefficient is greater for Mix 3 than for Mix 4. Thus, the enhancement in friction coefficient is a function of the specific crumb rubber particle area.

Nevertheless, rubber reinforcement adversely impacts the strength properties. As seen in Table 6, although the average value of the scratch hardness increases by 46% after addition of 10% wt microsilica, a subsequent decrease of 20% and 16% in scratch hardness is recorded after further addition of respectively 5% wt and 10% wt of crumb rubber particles. Similar results have been reported in the literature: a loss in compressive...
strength was observed after partial replacement of aggregates by crumb rubber in self-compacting concrete [14, 44]. Furthermore, the strength loss was positively correlated to the volume content of rubber [44]. However, in our case, due to imperfect bonding between the rubber particles and the surrounding hardened cement matrix, additional air voids were incorporated in the mix as seen in Fig. 1b). This increase in porosity $\Delta \phi$ due to improper bonding is proportional to the rubber particle size $r$ and the rubber volume content $\phi_r$: $\Delta \phi \propto 2\pi rt\phi_r$, where $t$ is the thickness of rubber particles. As a result, the relative increase in porosity due to improper bonding is 2.5 times greater for Mix 3 than for Mix 4. Therefore, the additional porosity due to improper bonding explains the slightly lower scratch hardness of Mix 3 compared to Mix 4. Nevertheless, the joint addition of fume silica and crumb-rubber contributes to shifting the frequency distribution curve towards high values.

The average fracture toughness $K_c$ increases by 29% by addition of microsilica, and by 38% and 12% after subsequent addition of respectively 5% and 10% crumb rubber. Our findings concur with that of other scientists who reported an en-

### Table 6: Influence of crumb rubber content and fume silica content on aggregate mechanical characteristics.

<table>
<thead>
<tr>
<th>Property</th>
<th>Mix 1</th>
<th>Mix 2</th>
<th>Mix 3</th>
<th>Mix 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>$H$, MPa</td>
<td>473.7</td>
<td>690.3</td>
<td>549.08</td>
<td>580.2</td>
</tr>
<tr>
<td>$\mu$</td>
<td>0.270</td>
<td>0.273</td>
<td>0.297</td>
<td>0.289</td>
</tr>
<tr>
<td>$K_c$, MPa $\sqrt{m}$</td>
<td>0.34</td>
<td>0.44</td>
<td>0.47</td>
<td>0.38</td>
</tr>
<tr>
<td>$\eta$</td>
<td>0.40</td>
<td>0.51</td>
<td>0.54</td>
<td>0.42</td>
</tr>
<tr>
<td>$\beta$</td>
<td>2.15</td>
<td>4.04</td>
<td>2.42</td>
<td>3.18</td>
</tr>
</tbody>
</table>

Fig. 7 displays the frequency distribution of the fracture toughness $K_c$ for all four materials. (Color online)
hancement in fracture resistance after addition of crumb rubber particles [12]. The gain in fracture resistance is commonly attributed to the intrinsic ductility of rubber particles as well as the presence of toughening mechanisms such as crack ligament bridging, which are promoted by the presence of rubber particles.

5.3. Influence of Surface Lubricant or Resistance to Weathering

In railway applications, a major concern is to appraise the durability of materials in harsh environmental conditions: wet due to water (rain, snow) or oil (leaking from an engine). Thus, we assessed the influence of lubricant on the surface properties via surface lubrication with oil and deionized water as per protocols P 2–3. Fig. 8 displays the distribution of the friction coefficient for all four mixes and for all three surface conditions: dry, wet with oil, and wet with water. In addition, cluster analysis was implemented to decompose the overall probability distribution of the friction coefficient as a weighted sum of individual Gaussian distributions [45, 46]. Herein, each single Gaussian distribution represents a specific friction micromechanism. Friction is a surface phenomena that results from the interlocking of surface asperities. At the microscopic and nanoscale, friction depends on a wide range of parameters such as asperity density, asperity radius of curvature, contact shear strength, contact junction plastic yield strength, etc. [47]. We opt for a discrete representation of this continuum of friction-inducing micromechanisms using cluster analysis and multivariate mixture analysis [45, 46]. Section 7 in the Appendix displays the weights and average friction coefficient of each individual micromechanisms, whereas the corresponding probability distribution curves are shown in Fig. 8.

Without crumb rubber, high-net-friction micromechanisms are curbed due to chemical reactivity. For instance, for the reference specimen, Mix 1 without crumb rubber, friction micromechanisms with a net average friction coefficient of 0.45 and above are drastically suppressed after surface wetting with oil or deionized water. This drastic reduction in high-net-friction mechanisms is even more noticeable for surface treatment with deionized water. A plausible reason is the interaction of water molecules with hardened cement paste. Surface water may activate a further hydration of cement paste, seep into the cement paste micropores and nanopores, locally increase the pore pressure and generate additional microcracking. As a result of the interaction between cement paste and water, local topological features such as asperities may be masked, resulting in a smoothing of the surface. A similar phenomenon is observed for Mix 2 (conc+silica) when the surface is wet with water. In this case, the water will contribute to sub-critical cracking of silica via stress corrosion cracking [48].

In contrast, crumb rubber inclusions promote the rise of high-net-friction micromechanisms. For Mix 3 (conc+silica+5% wt rubber), friction micromechanisms with a net average friction coefficient greater than 0.5 are still active in presence of oil or water. As a result, for Mix 3, higher values of the friction coefficient were recorded in presence of water and oil. One reason is the chemical inertia of rubber with respect to water and oil which contributes to an enhancement of local asperities. Mix 4 (conc+silica+10% wt rubber) experiences a sharper decrease of the friction coefficient when the surface is treated with oil. This might be due to the smaller specific surface area $R_a$ of the crumb rubber. Nevertheless, overall the partial replacement of aggregates with crumb rubber contributes to a higher resistance to weathering and an improved stability of surface friction properties with respect to surface treatment with a lubricant.

6. Conclusions

To understand the tribological behavior of crumb-rubber concrete, scratch testing has been applied at different length-scales,
Figure 8: Influence of lubricant on friction coefficient. (Color online)
and under different loading and speed rates, and for various surface treatments. Optical microscopy and scanning electron microscopy were utilized to identify the micro-constituents, whereas contact mechanics and fracture mechanics were utilized to yield the mechanical characteristics. Based on the testing results, the following conclusions can be derived:

1. Crumb rubber inclusions contribute to an increase in the effective friction behavior.

2. An enhancement of the fracture toughness is observed with the addition of crumb rubber particles.

3. A high resistance to weathering a higher stability in the tribological response with respect to surface lubrication is observed for crumb-rubber reinforced concrete.

4. The specific surface area of crumb rubber particles may plan a crucial roles in governing the level on improvement of the friction coefficient. In addition, the functionalization of the cement/rubber interface using bonding agent may stall the decrease in strength observed due to the partial replacement of aggregates with crumb rubber particles. Nevertheless, further research is needed.

Thus, these results will contribute to the development of enhanced-performance materials for railroad applications.

Acknowledgments

The research was funded by the Birmingham-Illinois Partnership for Discovery, Engagement and Education (BRIDGE). The research was also funded by the Start-Up funds of Prof. Akono that were provided jointly by the Department of Civil and Environmental Engineering as well as the College of Engineering at the University of Illinois at Urbana-Champaign. We are thankful to Caroline V. Johnson for the help in carrying out part of scratch friction tests. This research was carried out in part in the Frederick Seitz Materials Research Laboratory.

Central Research Facilities, University of Illinois, and was also partly carried out in part in the Imaging Technology Group at the Beckman Institute for Advanced Science and Technology at the University of Illinois.

References


36, pp. 617–622.

Hameed, Mechanical, Fracture, and Microstructural Investigations of

self compacting rubberized concrete, Construction and Building Materi-

[14] N. Ganesan, Bharati Raj J and A.P. Shashikala, Strength and durabil-
ity studies of self compacting rubberised concrete, The Indian Concrete

Tribology with Atomic Force Microscopy, Chemical Reviews, (1997), 57

[16] M. A. Lantz, S. J. O’Shea, A. C. F. Hoole, M. E. Welland, Lateral sti-
derstanding and quantification of elastic and plastic deformation during a

[17] Alva Peled, Jason Weiss, Hydrated cement paste constituents ob-
erved with Atomic Force and Lateral Force Microscopy, Construc-
tion and Building Materials, (2011), Vol. 25, pp. 4299–4302, DOI:

(AFM and LFM) examinations of cement and cement hydration prod-
ucts, Cement and Concrete Composites, (2013), Vol. 36, pp. 48–55, DOI:
10.1016/j.cemconcomp.2012.08.021.

[19] M. Jafarbeglou, M. Abdouss, A. A. Ramezanianpour, Nanoscience and
Nano Engineering in Concrete Advances A Review, Int. J. Nanosci. Nan-
otechnology, (2015), DOI: 10.1061/ASCE/NM.2153-
0040-6090(95)08138-0.

[20] Bull, S. J., Can scratch testing be used as a model for the abrasive wear

derstanding and quantification of elastic and plastic deformation during a

[22] Li. K and Shapiro, Y. and Li, J.-C.-M., Scratch test of soda-lime glass,

[23] Liang, Y.N. and Li, S.-Z. and Li, D.-F. and Li, S., Some developments for

[24] Low, S. R., Rockwell Hardness of Metallic Materials, National Insti-
tute of Standards and Technology, Special Publication, (2001), DOI:
10.1016/0003-4916(63)90068-X.

[25] Subhash, G. and Zhang, W., Investigation of the overall friction coeffi-

roughness criteria for cement paste nanoindentation, Cement and Con-

[27] Xu, J. and Yao, W., Nano-scratch as a new tool for assessing the nano-
tribological behavior of cement composite, Materials and Structures,


[29] ASTM, ASTM G171-03: Standard test method for scratch hardness
of materials using a diamond stylus1, ASTM International, West Con-
shohocken, PA (2009), DOI:10.1520/G0171-03.

[30] J.A. Williams, Analytical models of scratch hardness, Tribology Interna-

[31] Ange-Therese Akono, Nicholas X. Randall, Franz-Josef Ulm, Experiment-
damental determination of the fracture toughness via microscratch tests:
Application to polymers, ceramics and metals, Mat. Res. Soc., 27, (2012),
485-493


[33] Akono, A.-T., Energetic Size Effect Law at the Microscopic Scale: Appli-
cation to Progressive-load Scratch Testing, ASCE’s Journal of Nanome-
chanics and Micromechanics, (2015), DOI: 10.1061/(ASCE)NM.2153-
5477.0000105.

[34] Anderson, K. and Akono, A.T., 2017. Microstructure-toughness rela-
tionships in calcium aluminate cementpolymer composites using instru-
mented scratch testing. Journal of Materials Science, 52(22), pp.13120-
13132.


[37] Blushan, B. and Kulkarni, A.V., Effect of normal load on microscale fric-
10.1016/0040-6090(95)08138-0.

[38] Roth, F.-L. and Driscoll, R.-L. and Holt, W.-L., Frictional Properties of
Rubber, Rubber Chemistry and Technology, (1943), Vol. 16, pp. 155–177,
DOI:10.5254/1.3540095.

Law at the Microscopic Scale: Appli-
cation to polymers, ceramics and metals, Mat. Res. Soc., 27, (2012),
485-493

[40] Bouissou, S. and Petit, J.-P. and Barquins, M, Normal load, slip rate and
roughness influence on the polymethylmethacrylate dynamics of sliding


Appendix

7. Mixture Analysis of Friction Frequency Distribution: Effect of Surface Lubricant

Tables 7–10 below display the characteristics of the individual friction mechanisms identified for each mix and for each surface treatment condition. Three surface treatment were considered; Dry, wet with Oil and wet with Deionized Water. The individual friction mechanisms are characterized by their fraction, (%), average friction coefficient, $<\mu>$, and standard deviation of the friction coefficient, $<\sigma_\mu>$. 

### Table 7: Deconvolution analysis of the friction distribution for Mix 1.

<table>
<thead>
<tr>
<th>Mix 1</th>
<th>Mechanism</th>
<th>Mechanism</th>
<th>Mechanism</th>
<th>Mechanism</th>
<th>Mechanism</th>
<th>Mechanism</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
</tr>
<tr>
<td>Dry</td>
<td>(%)</td>
<td>0.43</td>
<td>0.11</td>
<td>0.10</td>
<td>0.20</td>
<td>0.11</td>
</tr>
<tr>
<td></td>
<td>$\mu$</td>
<td>0.16</td>
<td>0.24</td>
<td>0.30</td>
<td>0.38</td>
<td>0.47</td>
</tr>
<tr>
<td></td>
<td>$\sigma_\mu$</td>
<td>0.03</td>
<td>0.03</td>
<td>0.03</td>
<td>0.05</td>
<td>0.04</td>
</tr>
<tr>
<td></td>
<td>(%)</td>
<td>0.59</td>
<td>0.05</td>
<td>0.07</td>
<td>0.18</td>
<td>0.05</td>
</tr>
<tr>
<td>Oil</td>
<td>$\mu$</td>
<td>0.18</td>
<td>0.25</td>
<td>0.30</td>
<td>0.38</td>
<td>0.47</td>
</tr>
<tr>
<td></td>
<td>$\sigma_\mu$</td>
<td>0.03</td>
<td>0.02</td>
<td>0.02</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td></td>
<td>(%)</td>
<td>0.58</td>
<td>0.05</td>
<td>0.07</td>
<td>0.15</td>
<td>0.09</td>
</tr>
<tr>
<td>DI Water</td>
<td>$\mu$</td>
<td>0.10</td>
<td>0.18</td>
<td>0.24</td>
<td>0.32</td>
<td>0.42</td>
</tr>
<tr>
<td></td>
<td>$\sigma_\mu$</td>
<td>0.03</td>
<td>0.02</td>
<td>0.03</td>
<td>0.05</td>
<td>0.05</td>
</tr>
</tbody>
</table>

### Table 8: Deconvolution analysis of the friction distribution for Mix 2.

<table>
<thead>
<tr>
<th>Mix 2</th>
<th>Mechanism</th>
<th>Mechanism</th>
<th>Mechanism</th>
<th>Mechanism</th>
<th>Mechanism</th>
<th>Mechanism</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
<td>5</td>
<td>6</td>
</tr>
<tr>
<td>Dry</td>
<td>(%)</td>
<td>0.41</td>
<td>0.13</td>
<td>0.13</td>
<td>0.23</td>
<td>0.05</td>
</tr>
<tr>
<td></td>
<td>$\mu$</td>
<td>0.15</td>
<td>0.24</td>
<td>0.31</td>
<td>0.41</td>
<td>0.49</td>
</tr>
<tr>
<td></td>
<td>$\sigma_\mu$</td>
<td>0.05</td>
<td>0.04</td>
<td>0.03</td>
<td>0.05</td>
<td>0.04</td>
</tr>
<tr>
<td></td>
<td>(%)</td>
<td>0.30</td>
<td>0.05</td>
<td>0.12</td>
<td>0.43</td>
<td>0.05</td>
</tr>
<tr>
<td>Oil</td>
<td>$\mu$</td>
<td>0.11</td>
<td>0.19</td>
<td>0.25</td>
<td>0.35</td>
<td>0.49</td>
</tr>
<tr>
<td></td>
<td>$\sigma_\mu$</td>
<td>0.04</td>
<td>0.02</td>
<td>0.04</td>
<td>0.06</td>
<td>0.04</td>
</tr>
<tr>
<td></td>
<td>(%)</td>
<td>0.54</td>
<td>0.09</td>
<td>0.15</td>
<td>0.08</td>
<td>0.09</td>
</tr>
<tr>
<td>DI Water</td>
<td>$\mu$</td>
<td>0.16</td>
<td>0.25</td>
<td>0.34</td>
<td>0.41</td>
<td>0.48</td>
</tr>
<tr>
<td></td>
<td>$\sigma_\mu$</td>
<td>0.03</td>
<td>0.04</td>
<td>0.04</td>
<td>0.03</td>
<td>0.04</td>
</tr>
<tr>
<td>Mix 3</td>
<td>Mechanism 1 (%)</td>
<td>Mechanism 2 (%)</td>
<td>Mechanism 3 (%)</td>
<td>Mechanism 4 (%)</td>
<td>Mechanism 5 (%)</td>
<td>Mechanism 6 (%)</td>
</tr>
<tr>
<td>-------</td>
<td>----------------</td>
<td>----------------</td>
<td>----------------</td>
<td>----------------</td>
<td>----------------</td>
<td>----------------</td>
</tr>
<tr>
<td>Dry</td>
<td>0.45</td>
<td>0.07</td>
<td>0.12</td>
<td>0.18</td>
<td>0.10</td>
<td>0.07</td>
</tr>
<tr>
<td></td>
<td>&lt;μ&gt;</td>
<td>0.12</td>
<td>0.23</td>
<td>0.31</td>
<td>0.41</td>
<td>0.49</td>
</tr>
<tr>
<td></td>
<td>σμ</td>
<td>0.05</td>
<td>0.03</td>
<td>0.04</td>
<td>0.04</td>
<td>0.03</td>
</tr>
<tr>
<td></td>
<td>(%)</td>
<td>0.15</td>
<td>0.16</td>
<td>0.15</td>
<td>0.09</td>
<td>0.37</td>
</tr>
<tr>
<td>Oil</td>
<td>0.12</td>
<td>0.21</td>
<td>0.29</td>
<td>0.36</td>
<td>0.46</td>
<td>0.58</td>
</tr>
<tr>
<td></td>
<td>&lt;μ&gt;</td>
<td>0.48</td>
<td>0.05</td>
<td>0.09</td>
<td>0.14</td>
<td>0.18</td>
</tr>
<tr>
<td></td>
<td>σμ</td>
<td>0.04</td>
<td>0.03</td>
<td>0.04</td>
<td>0.03</td>
<td>0.06</td>
</tr>
<tr>
<td></td>
<td>(%)</td>
<td>0.48</td>
<td>0.05</td>
<td>0.09</td>
<td>0.14</td>
<td>0.18</td>
</tr>
<tr>
<td>DI Water</td>
<td>0.18</td>
<td>0.28</td>
<td>0.35</td>
<td>0.44</td>
<td>0.53</td>
<td>0.72</td>
</tr>
<tr>
<td></td>
<td>&lt;μ&gt;</td>
<td>0.04</td>
<td>0.03</td>
<td>0.04</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td></td>
<td>σμ</td>
<td>0.04</td>
<td>0.03</td>
<td>0.04</td>
<td>0.05</td>
<td>0.05</td>
</tr>
</tbody>
</table>

Table 9: Deconvolution analysis of the friction distribution for Mix 3.

<table>
<thead>
<tr>
<th>Mix 4</th>
<th>Mechanism 1 (%)</th>
<th>Mechanism 2 (%)</th>
<th>Mechanism 3 (%)</th>
<th>Mechanism 4 (%)</th>
<th>Mechanism 5 (%)</th>
<th>Mechanism 6 (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry</td>
<td>0.38</td>
<td>0.09</td>
<td>0.16</td>
<td>0.26</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td></td>
<td>&lt;μ&gt;</td>
<td>0.14</td>
<td>0.22</td>
<td>0.31</td>
<td>0.44</td>
<td>0.55</td>
</tr>
<tr>
<td></td>
<td>σμ</td>
<td>0.05</td>
<td>0.04</td>
<td>0.05</td>
<td>0.06</td>
<td>0.05</td>
</tr>
<tr>
<td></td>
<td>(%)</td>
<td>0.43</td>
<td>0.14</td>
<td>0.13</td>
<td>0.11</td>
<td>0.12</td>
</tr>
<tr>
<td>Oil</td>
<td>0.12</td>
<td>0.22</td>
<td>0.31</td>
<td>0.39</td>
<td>0.49</td>
<td>0.63</td>
</tr>
<tr>
<td></td>
<td>&lt;μ&gt;</td>
<td>0.04</td>
<td>0.04</td>
<td>0.03</td>
<td>0.03</td>
<td>0.04</td>
</tr>
<tr>
<td></td>
<td>σμ</td>
<td>0.04</td>
<td>0.03</td>
<td>0.04</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td></td>
<td>(%)r</td>
<td>0.56</td>
<td>0.05</td>
<td>0.10</td>
<td>0.16</td>
<td>0.06</td>
</tr>
<tr>
<td>DI Water</td>
<td>0.16</td>
<td>0.24</td>
<td>0.31</td>
<td>0.40</td>
<td>0.51</td>
<td>0.64</td>
</tr>
<tr>
<td></td>
<td>&lt;μ&gt;</td>
<td>0.04</td>
<td>0.03</td>
<td>0.04</td>
<td>0.05</td>
<td>0.05</td>
</tr>
</tbody>
</table>

Table 10: Deconvolution analysis of the friction distribution for Mix 4.