

表面摩擦磨损与润滑

## 金属铝表面超疏水薄膜的构筑及减摩特性

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**摘要:**目的 改善铝基材料在干摩擦条件下的摩擦磨损性能。方法 采用两步法在铝表面构筑稳定的超疏水薄膜。首先采用盐酸溶液刻蚀金属铝,在其表面构筑微纳结构;然后涂覆硬脂酸,降低表面能。利用 SEM、XRD、FTIR、接触角测量仪及摩擦磨损试验机表征铝表面超疏水薄膜的表面形貌、化学组分、润湿性和减摩耐磨特性。结果 SEM 及 XRD 分析表明,刻蚀后的铝表面呈现多尺度微纳结构。FTIR 分析表明,脂肪酸以双配位结构与铝表面发生作用。接触角测试表明,所制备的薄膜呈现出良好的超疏水性,静态接触角达  $150^\circ$ ,滑动角小于  $10^\circ$ 。摩擦学实验结果表明,制备的超疏水薄膜可明显改善铝基底的摩擦学性能,在干摩擦条件下与钢球对磨时,超疏水薄膜的摩擦系数保持在 0.16 左右,寿命超过 10 000 s,而相同条件下未处理的金属铝摩擦系数超过 0.6。结论 采用盐酸溶液刻蚀金属铝,然后涂覆硬脂酸,可在铝表面构筑复合薄膜。薄膜不仅表现出明显的超疏水特性,同时具有良好的减摩耐磨性能。该方法技术简单,价格低廉,易于批量化生产,为改善微纳条件下铝及其合金的摩擦学性能提供了一个新的思路。

**关键词:**超疏水;铝;减摩;化学刻蚀;硬脂酸

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## Fabrication and Friction-reducing Performance of Superhydrophobic Film on Aluminum

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**ABSTRACT:** **Objective** To improve the friction and wear performance of aluminum in dry sliding contact. **Methods** The two-step method was used in this paper to fabricate the superhydrophobic film on aluminum. The aluminum substrate was firstly etched by hydrochloric acid to create the micro- and nano-texture, then coated by layer of stearic acid to decrease surface energy. SEM, XRD, FTIR spectroscopy, contact-angle measuring instrument and friction-abrasion testing machine were used to analyze the morphological features, chemical composition, hydrophobicity and tribological performance of superhydrophobic film on aluminum, respectively. **Results** SEM and XRD analyses showed that the micro- and nano-structure was created on aluminum after chemical

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etch. FTIR analysis showed stearic acid was chemically absorbed on aluminum surface in a bidentate mode. The film on aluminum showed superhydrophobicity with static water contact angle of  $150^\circ$  and sliding angle of  $10^\circ$ . It was effective to improve tribological performance of aluminum. The friction coefficient kept as low as 0.16 for 10 000 s sliding time when sliding with steel ball in the unlubricated condition. **Conclusion** The superhydrophobic film formed on aluminum by using chemical etch by hydrochloric acid and then the surface modified by stearic acid could improve the friction and wear performance of aluminum. The method is simple and can be easily operated at a large scale. It may provide a new method to improve the friction and wear performance of aluminum.

**KEY WORDS:** superhydrophobicity; aluminum; friction-reducing performance; chemical etch; stearic acid

近年来,超疏水材料以独特的润湿性能引起了人们的广泛兴趣,已成为材料科学中研究的热点课题之一<sup>[1-5]</sup>。水无法浸润超疏水表面,而是以液滴的形式存在,这极大地减小了水与材料表面的直接接触,因此在金属基体上构筑的超疏水表面表现出极佳的耐腐蚀性能<sup>[6]</sup>。同时,超疏水表面还具有自清洁性能、抗结霜性能和界面减阻效应<sup>[7]</sup>。已有研究表明,固体表面的润湿性由表面化学成分和表面粗糙度共同决定。其中前者决定表面呈亲水性质,还是呈疏水性质;后者的作用犹如放大器,亲水的表面经织构化处理后将更加亲水,而疏水表面经织构化处理后将更加疏水。由于目前使用的材料大多表面本身呈亲水性,因此通过在表面构筑微纳织构无法实现疏水性。人们通过对自然界中多种植物叶子、昆虫翅膀以及水鸟羽毛等生物表面进行研究,发现了许多在材料基体上构筑超疏水表面的方法<sup>[8-13]</sup>,如气相沉积法、电化学方法、溶胶凝胶法等等。究其根本,要实现超疏水表面一般需要两步完成:首先是在表面构筑适宜的微纳结构,然后在微纳结构表面涂覆低表面能物质。其中,第一步是构筑超疏水表面不可缺少的步骤。

相比于其他金属材料,铝及其合金比重小、质量轻,并且具有良好的延展性和可塑性,广泛应用于日常生活和工业生产中。然而,铝合金耐磨和抗擦伤性能差,特别是在钢-铝接触条件下,易发生铝表面向钢对偶的转移,造成铝件严重磨损,这种情况在铝合金的加工过程中尤为严重。铝向钢的转移会严重缩短模具的使用寿命,极大地影响铝工件的表面质量。因此,铝合金的润滑一直是困扰摩擦学研究工作者的难题之一<sup>[14-17]</sup>。近年来,随着铝基材料在数字微镜器件中的应用<sup>[18-19]</sup>,铝合金在微纳米接触条件下的润滑问题开始引起人们的关注。目前,多采用自组装单分子膜成膜技术在表面沉积疏水薄膜来减小微纳尺度下铝基材料的摩擦、黏着和磨损<sup>[20-21]</sup>,但这类薄膜耐磨性能差,使用寿命短,无法满足微型机械连续润滑的要求。

近年来,一些研究者开始尝试在金属材料上构筑

超疏水表面达到减摩和耐磨的目的。Alpas等<sup>[22]</sup>制备了具有荷叶表面形貌的微/纳尺度仿生织构镍表面,表面的接触角可达 $156^\circ$ ,具有明显的减摩效应。笔者所在项目组在前期工作中也曾发现<sup>[23]</sup>,45#钢表面的超疏水薄膜具有优异的减摩和耐磨特性。基于此,文中尝试在金属铝表面构筑超疏水薄膜,表征了超疏水薄膜的摩擦学特性,以期改善微纳尺度下铝基材料的摩擦学性能提供一些借鉴。

## 1 试验

### 1.1 铝表面超疏水薄膜的制备

铝基体尺寸为 $15\text{ mm}\times 15\text{ mm}$ ,依次用400,800,1500目砂纸打磨抛光至镜面,再用去离子水和无水乙醇依次超声处理10 min,最后用氮气吹干。

配制2 mol/L盐酸溶液作为刻蚀液。制备步骤如下:1)将清洗后的铝试块浸泡在 $95^\circ\text{C}$ 的刻蚀液中,保持30 s;2)取出后,用去离子水和无水乙醇依次超声清洗,并用氮气吹干;3)立即浸入8%的水合肼溶液中,于 $25^\circ\text{C}$ 保持24 h;4)取出后,浸入0.01 mol/L的硬脂酸乙醇溶液中,于 $25^\circ\text{C}$ 放置24 h;5)取出后,用无水乙醇清洗,并用氮气吹干。为作比较,采用同样的方法在未经刻蚀处理的铝表面沉积了硬脂酸薄膜。

### 1.2 铝表面超疏水薄膜的性能测试

1)采用CAM101接触角测定仪测量样品的水接触角。在样品表面任意选取5个位置进行测量,取平均值。

2)采用D/MAX-RB型X射线衍射仪表征涂层的晶体结构,采用Hitachi S-3500N型扫描电子显微镜观察铝刻蚀前后的表面形貌。

3)采用Nicolette iN10傅里叶变换红外光谱仪对硬脂酸薄膜的成键特征进行分析。

4)利用UMT-3型摩擦磨损试验机测定超疏水薄膜的摩擦学性能。对偶件为 $\phi 4$ 轴承钢球,采用往复形式,行程6 mm,频率2 Hz,施加载荷0.1 N。

## 2 结果及讨论

### 2.1 表面物相及形貌

图1为金属铝表面经2 mol/L 盐酸溶液化学刻蚀前后的XRD图谱。可以看出,两个样品呈现完全相同的衍射图谱, $2\theta$ 为 $38.2^\circ$ ,  $44.6^\circ$ ,  $65.0^\circ$ ,  $78.1^\circ$ 的衍射峰分别归结为铝的(111), (200), (220), (311)晶面,无其他衍射峰出现。这说明金属铝经化学刻蚀后,表面未产生新的物相。

图2为金属铝表面刻蚀前后的SEM图片。图2a

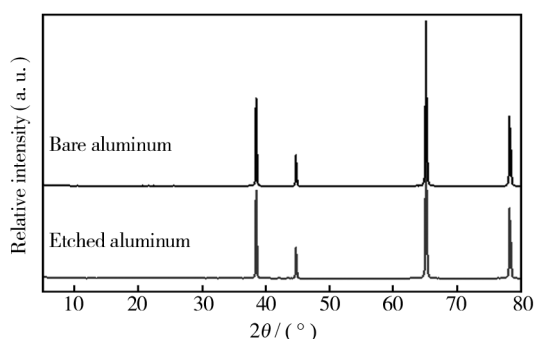


图1 金属铝化学刻蚀前后的XRD谱图

Fig. 1 XRD spectra of aluminum before and after chemical etch

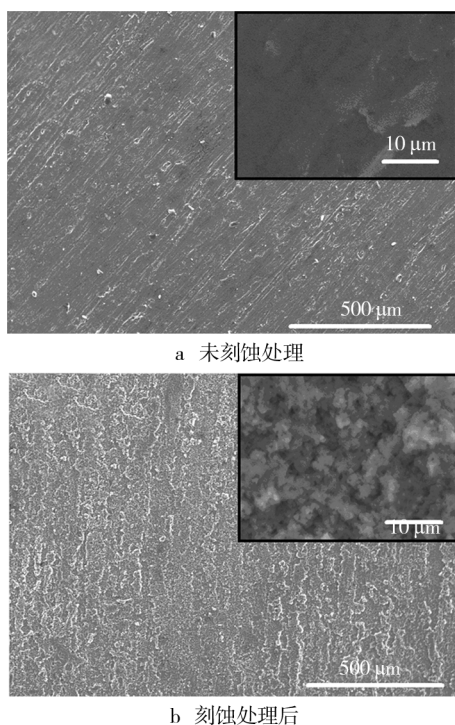


图2 金属铝刻蚀前后的表面形貌

Fig. 2 SEM images of the aluminum before and after chemical etch; a) the bare aluminum, b) the etched aluminum

显示,刻蚀前的铝表面仅存在一些加工痕迹,相对光滑。图2b显示,刻蚀后的铝表面由原始的平滑状纹理变成了均匀的颗粒状,且颗粒状之间存在微小的缝隙,呈现多尺度微纳结构。

### 2.2 水接触角

在未刻蚀及经过刻蚀的金属铝表面沉积硬脂酸薄膜,并对其润湿性能进行表征,图3为沉积硬脂酸薄膜前后的润湿性测定结果。可以看出,未刻蚀金属铝表面对水的接触角接近 $80^\circ$ ,沉积硬脂酸薄膜后,对水的接触角增大到 $108^\circ$ ;刻蚀金属铝表面对水的接触角仅为 $5^\circ$ ,表现出超亲水特性,沉积硬脂酸薄膜后,对水的接触角达到 $150^\circ$ ,同时水滴在表面很难停留,滚动角小于 $10^\circ$ 。大的静态接触角和小的滚动角表明,在刻蚀后的金属铝基体表面沉积的硬脂酸薄膜处于超疏水状态。根据Cassie-Baxter公式<sup>[24]</sup>可算得固-液界面所占的比例约为0.15,说明经化学刻蚀后,铝表面存在的多尺度微纳结构截留的空气可能阻止了水滴渗透到沟槽内,使其仅停留在结构表面,从而显著增大了表面对水的接触角。

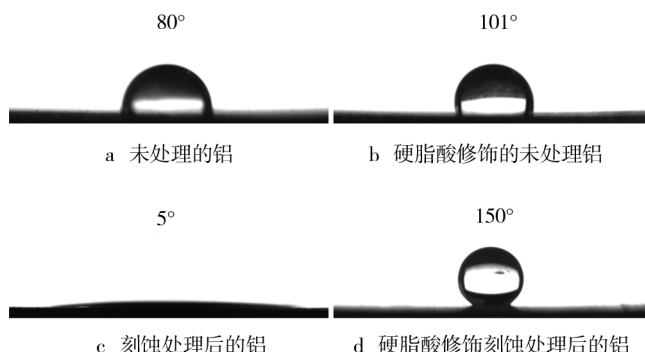


图3 样品表面静态水接触角图像

Fig. 3 Water droplet images for samples: a) nonetched aluminum, b) stearic acid film on nonetched aluminum, c) etched aluminum, d) stearic acid film on etched aluminum

### 2.3 薄膜成键特性

图4为未刻蚀铝和刻蚀铝表面沉积的硬脂酸薄膜的IR谱图。不难看出,两种薄膜均在 $2914\text{ cm}^{-1}$ 处出现了吸收峰。事实上,如果脂肪酸在金属表面形成有序薄膜(烷基链呈全反式结构,并都以同一角度向表面倾斜),必有 $<2918\text{ cm}^{-1}$ 的特征吸收峰。因此认为,硬脂酸在未刻蚀铝和刻蚀铝表面均形成了有序薄膜。在C—O振动区,与固态硬脂酸的IR谱图相比,



1700  $\text{cm}^{-1}$  处的吸收峰(对应于硬脂酸的自由羧基)不再存在, C=O 和 C—O 键的振动峰位分别偏移到 1584.8  $\text{cm}^{-1}$  和 1471.5  $\text{cm}^{-1}$  处。这说明在金属铝表面沉积的硬脂酸薄膜中硬脂酸的端基不再呈 COOH 结构, 而可能以双配位结构与铝表面发生了作用<sup>[25]</sup>。同时观察到, 相比之下, 未刻蚀铝表面的硬脂酸薄膜 IR 吸收峰强度较低。这可能是因为刻蚀后的铝表面为高亲水性的羟基化表面, 硬脂酸易于吸附其上, 并且铝刻蚀后的沟壑状结构也为硬脂酸分子提供了更大的吸附反应面积, 从而有助于形成更为致密的硬脂酸薄膜。

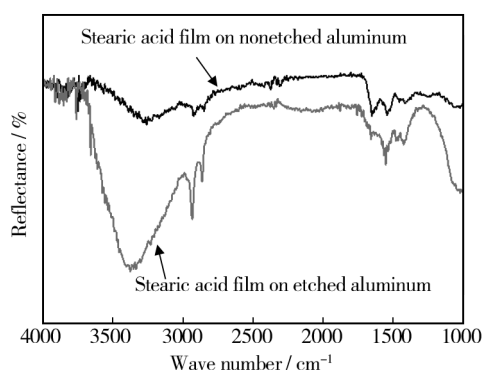


图4 硬脂酸薄膜的红外谱图

Fig. 4 FT-IR spectra of stearic acid films

## 2.4 薄膜摩擦学性能

对铝表面硬脂酸薄膜的摩擦学性能进行了表征, 并与未刻蚀铝进行对比, 摩擦系数随滑动时间的变化如图5所示。可以看出, 磨合后, 金属铝的摩擦系数达到0.6左右, 而且波动较大。在铝表面直接沉积的硬脂酸薄膜对铝起到一定的保护效果, 磨合后的摩擦系数保持在0.5左右。在经化学刻蚀的铝表面沉积的硬脂酸超疏水薄膜则表现出十分优异的减摩性能, 摩擦系数一直保持0.16左右。

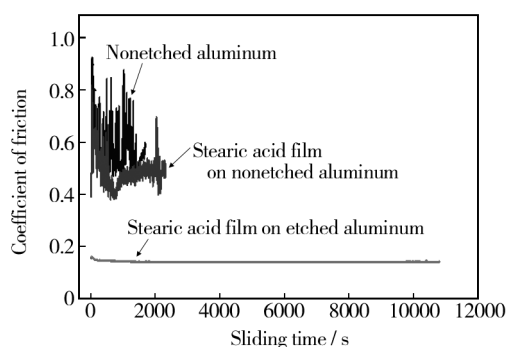


图5 摩擦系数-滑动时间关系曲线

Fig. 5 The relationship of coefficient of friction and sliding time

铝表面超疏水薄膜的优异减摩性能无疑与其独特的润湿性和结构有关。已经证实, 在干摩擦条件下, 真实接触面积的大小直接影响摩擦力的高低<sup>[26]</sup>, 而真实接触面积不仅仅取决于正压力, 更是与界面能直接相关<sup>[27]</sup>。材料表面能的大小可用表面静态接触角的余弦值衡量<sup>[28]</sup>, 接触角越大说明表面能越低, 这可能就是实验中铝表面的超疏水薄膜表现出极低摩擦系数的主要原因<sup>[29-30]</sup>。另外, IR 分析发现, 在经化学刻蚀的铝表面涂覆的硬脂酸薄膜具有更加致密的结构, 因而表现出持久、稳定的减摩耐磨效果<sup>[31]</sup>。

## 3 结论

1) 利用盐酸溶液对金属铝进行刻蚀后, 铝表面形成了特殊的微纳结构, 在结构表面沉积的硬脂酸薄膜具有超疏水性能, 对水的接触角达 150°, 滑动角小于 10°。

2) 铝表面超疏水薄膜具有优异的减摩耐磨性能, 摩擦系数保持在 0.16 左右, 耐磨寿命超过 10 000 s; 而未经处理的铝表面经过短暂磨合后, 摩擦系数超过 0.6。超疏水薄膜优异的减摩耐磨性能可能与其极低的表面能有关, 同时相对致密的结构也对其减摩耐磨性能有所贡献。

3) 采取简单的化学刻蚀及硬脂酸改性在金属铝表面构筑的超疏水薄膜具有极为优异的减摩及耐磨性能。该方法技术简单, 价格低廉, 易于批量化生成, 为改善微纳条件下铝及其合金的摩擦学性能提供了一个新的思路。

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