

专题—超润滑表面工程

醇分子吸附对石墨基面纳米摩擦行为的影响研究

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摘要: **目的** 探究石墨基面的纳米尺度摩擦行为及其调控机制。**方法** 将表面洁净的高定向热解石墨块状晶体浸没在高纯度的醇中, 一段时间后取出并用干燥氮气吹干。用原子力显微镜对石墨基面上的摩擦力和黏附力进行测量。**结果** 石墨基面的摩擦力和黏附力在醇中处理后发生显著变化, 且随着醇分子碳链的增长而减小, 石墨基面上的超低摩擦在正庚醇中处理后减少了约 50%, 而在甲醇中处理后则增加了 8 倍。研究表明, 吸附在接触界面的醇分子能够调节相对滑动界面的接触公度性, 构象熵大的高碳醇分子促进了界面滑动从而减小摩擦力。接触区域发生的摩擦化学反应也是造成石墨表面摩擦力变化的重要原因, 反应产物的生成增大了探针与石墨之间的接触面积, 从而增大摩擦力。此外, 摩擦化学反应速率随着醇分子碳链长度的增加而减慢, 进一步影响了摩擦性能。**结论** 本研究揭示了通过物理吸附醇分子来调节石墨表面摩擦学性能的新机制, 结果表明醇类分子的结构和摩擦化学反应对润滑性能有显著影响, 这一发现为调控石墨以及石墨烯的摩擦性能提供了新思路。

关键词: 石墨; 醇分子吸附; 纳米摩擦; 摩擦化学反应; 原子力显微镜

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Effect of Alcohol Molecules Adsorption on Nanoscale Friction Behaviour of Graphite Basal Plane

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ABSTRACT: The work aims to investigate the nanoscale friction behaviour of graphite basal planes, as well as the mechanisms underlying its modulation. Highly oriented pyrolytic graphite (HOPG) crystals with freshly cleaved surfaces were immersed in high-purity alcohols for a controlled duration. After immersion, the crystals were dried with dry nitrogen gas to eliminate residual solvents for further analysis. The friction and adhesion forces on the graphite basal planes were then analyzed by atomic force microscopy (AFM). It was observed that both the friction and adhesion forces on the graphite surface notably changed

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following exposure to different alcohols. Specifically, the friction and adhesion forces decreased as the carbon chain length of the alcohol molecules increased. This result suggests a strong correlation between the molecular structure of alcohols and their ability to modulate the tribological properties of graphite basal planes. Under an applied load of 5 nN, the initially ultra-low friction on the graphite basal plane ((0.030 ± 0.024) nN) was reduced by approximately 50% ((0.014 ± 0.009) nN) after treatment in n-heptanol, a long-chain alcohol. In stark contrast, treatment with methanol, a short-chain alcohol, caused an eightfold increase in friction ((0.237 ± 0.024) nN). This striking variation in friction behaviour highlights the significant effect of alcohol chain length on the performance of sliding interface. The underlying mechanism for this modulation lies in the interaction between the alcohol molecules and the contact interface. Alcohol molecules adsorbed at the sliding interface are able to regulate the contact quality. Long-chain alcohols, such as n-heptanol, possess higher conformational entropy, which facilitates easier interfacial sliding, thereby reducing friction. In contrast, short-chain alcohols like methanol induce less conformational entropy and thus hinder the sliding process, leading to increased frictional resistance. Another crucial aspect of this study is the role of friction-induced chemical reactions. The Si atoms in the SiO_2 tip are highly susceptible to attack by the oxygen atoms in alcohol molecules, triggering nucleophilic substitution reactions, resulting in the formation of silica gel on the surface of the tip. The presence of this silica gel increases the contact area between the AFM tip and the graphite surface, which in turn contributes to higher friction and adhesion forces. Moreover, with the increase of the size of alcohol molecules, the nucleophilicity decreases, and the low carbon alcohol molecules are easier to approach silicon atoms in the process of nucleophilic substitution, and the tribochemical reaction rate is faster, which further affects the frictional behaviour. This study reveals a novel mechanism for regulating the frictional properties of graphite through the physical adsorption of alcohol molecules and friction-induced chemical reactions. The results demonstrate that the molecular structure of alcohols, particularly the length of their carbon chains, along with the rate of tribochemical reactions, play a critical role in determining the lubrication performance of graphite. These findings provide insights into the control and optimization of tribological properties of graphite, offering new opportunities for its application in nanoscale devices and lubrication systems.

KEY WORDS: graphite; alcohol molecules adsorption; nanoscale friction; tribochemical reactions; atomic force microscopy

摩擦是日常生活中普遍存在的现象,它贯穿于人们的生产和生活中。摩擦会导致能源的消耗。据统计,全球约有三分之一的一次能源是在摩擦过程中消耗的,摩擦及其引发的磨损也是导致 80% 机械零部件失效的主要原因^[1-2]。为了减小摩擦,人们开发了许多润滑方法,按照润滑剂的不同可以分为脂润滑、油润滑和固体润滑等。近年来,二维材料因其优秀的润滑性能引起了广泛关注,特别是石墨烯这一代表性的二维材料,作为一种优秀的固体润滑剂,在汽车工业^[3-4]、电子器件^[5-8]、航空航天^[9-10]、能源工业^[11-13]等多个领域得到了广泛应用。

随着对石墨烯润滑性能研究的深入,人们发现纳米尺度上石墨烯表面并不始终保持超低摩擦,而是会受到多种因素(如基底材料、气体环境、电场等)的影响^[14-24]。Li 等^[16]研究了各种基底上石墨烯表面的摩擦学性能,发现使用高表面能或表面光滑的材料作为基底时,石墨烯表面的摩擦力显著减小。大量研究表明,不同气体环境中石墨烯的摩擦学性能表现出显著差异,在干燥氮气环境中,石墨烯基面上表现出超低摩擦,而在潮湿环境中,水分子和氧气的存在严重破坏了石墨烯的润滑性能,摩擦力随着相对湿度的增加而增加^[17-19]。基于 C-AFM 技术,Lang 等^[20]发现石墨烯的摩擦学性能会受到电场的严重影响,在电场

环境中,石墨烯表面易发生电化学氧化并导致摩擦力变大。

基于对石墨烯在各种环境下摩擦行为变化的研究,衍生出一系列石墨烯纳米尺度摩擦学性能的调控手段。Chen 等^[25-26]提出了一种通过改变环境气体来调节石墨烯基面上超润滑行为的方法,发现具有不同分子结构的环境气体进入滑动界面后会导致不同的接触公度性,在苯酚气体环境中,由于苯酚的碳环与石墨烯的正六边形晶格相似,因此吸附在二氧化硅针尖表面的苯酚分子增加了针尖与石墨烯之间的公度性,从而导致超润滑行为的消失;而在正戊醇气体环境中,具有高构象熵的正戊醇分子吸附在二氧化硅表面进一步促进了界面滑动,从而增强了超润滑性。除此之外,Lang 等^[27]发现在导电针尖与石墨烯之间施加电压可以增加石墨烯表面的摩擦力,由此提出一种基于电化学氧化的石墨烯表面摩擦力的调控方法。Zeng 等^[28]通过等离子体处理来增强石墨烯表面的亲水性和结构缺陷的产生,以此来调控石墨烯的纳米尺度摩擦行为。由此可见,目前已经发展出许多对石墨烯表面摩擦学性能的调控方法,但这些方法都有一定的局限性,气体环境调控方法需要持续性的气氛控制,而电场和表面修饰调控方法都会不同程度地破坏石墨烯的原子结构,因此简易、无破坏性的石墨烯表

面摩擦调控方法还有待进一步丰富和拓展。

在本文中,提出了一种新的石墨纳米尺度摩擦行为的调控方法,基于 AFM 技术,测量了在不同碳链长度醇中处理过后石墨基面上的摩擦力和黏附力,研究表明,通过在不同碳链长度醇中的处理,可以增强或抑制石墨基面上的超润滑性,这种摩擦行为的改变是由于接触公度性的改变和摩擦化学反应引起的。

1 试验

1.1 石墨样品处理方法

测试前对样品的处理过程如图 1 所示,其中高定向热解石墨(HOPG)购自江苏先丰纳米材料科技有限公司。在空气中,对 HOPG 块状晶体的上下 2 个表面进行胶带剥离来得到洁净的石墨样品。随后,将石墨样品转移到醇(甲醇、正丙醇、正戊醇、正庚醇,纯度 $\geq 99.0\%$)中浸泡 5 min 后取出,用干燥氮气吹干后,装夹在原子力显微镜(AFM)测试平台上进行纳米尺度摩擦测试。

1.2 纳米尺度摩擦测试方法

在室温(约 25 °C)、干燥氮气气氛环境中,采用原子力显微镜(NX10, 韩国 Park 公司)测试样品表面的摩擦性能。试验中选用 Si 探针(Contact-G, 保加利亚 BudgetSensors 公司,标称弹性系数 0.2 N/m,标称针尖半径 8 nm)在接触模式下进行纳米摩擦试验,其表面覆盖有氧化层。AFM 探针的悬臂梁弹性系数按照 Sader 法进行校准,校准后弹性系数为 0.182 N/m。利用改进楔形法对 AFM 摩擦力进行标定,试验中使用楔形角为 70°的硅光栅(TGG1, Tipsnano, Estonia),并且为了尽量减小探针的磨损,在正戊醇气体环境中进行标定试验^[29]。在摩擦测试前,将 AFM 探针在紫外臭氧清洗机中处理 15 min,以去除可能存在的有机污染物。在测试过程中,选择测试区域时避免石墨台阶或褶皱等结构缺陷,将扫描范围选定在平坦的石墨基面上,AFM 探针的往复频率为 2 Hz,行程长度为 100 nm,则滑动速度为 400 nm/s,在同一位置重复扫描 128 次,将其记为 1 组测试,先后在 AFM 探针上施加 5、10、15 nN 的载荷,每个载荷下进行 3 组重复测试,测试过程中记录不同载荷下的摩擦力大小。在黏附力测试过程中,AFM 探针接近并接触样品,在探针上施加的纵向载荷从 0 nN 逐渐增加到 15 nN,

而后探针缩回并离开样品,通过分析力-距离($F-D$)曲线,计算黏附力大小。在同一样品表面随机选取 3 处平坦区域进行摩擦力和黏附力测试。

2 结果及分析

图 2 展示了 AFM 探针在扫描过程中的摩擦力信号,每条曲线是同一扫描位置处 128 次重复扫描的平均数据。每幅图中的蓝色和红色曲线代表了不同扫描方向扫描的数据,蓝色表示从左向右扫描,红色表示从右向左扫描,因此 2 条曲线符号相反。值得一提的是,本试验中选用的醇均为正醇,以确保碳链长度是唯一变量。不同醇中处理过的石墨表现出不同的摩擦力水平,碳链越长,摩擦力越小,这种规律并不受载荷变化的影响。而随着载荷的增大,摩擦力信号显著增大。

图 3 中展示了石墨表面经过不同醇处理之后的摩擦力。摩擦力是将 2 个方向的摩擦力信号相减后取平均值得到的。可以发现,随着醇从甲醇换到正庚醇,摩擦力显著减小。而使用相同探针在新撕开且未处理过的石墨表面测得的平均摩擦力分别为(0.030 \pm 0.024)、(0.044 \pm 0.024)、(0.076 \pm 0.049) nN,分别对应于 5、10、15 nN 的载荷。可以发现,当醇的碳链较短时,石墨表面摩擦力比未处理过的更大,如甲醇中处理过的石墨表面摩擦力增加了 8 倍,而正庚醇中处理过的石墨表面摩擦力减小了一半,说明醇分子的存在严重影响了滑动界面的接触和滑移情况。

图 4 展示了样品表面黏附力的测量结果。可以发现,不同样品表面的黏附力表现出明显差异,随醇的变化规律与横向力信号相似,随着碳链的增长而减小。在测试前和相邻 2 个样品的测试环节之间均在新撕开且未处理过的石墨表面测量 AFM 探针的黏附力,测量值始终保持稳定,大小为(3.74 \pm 0.51) nN,表明探针并未受到严重磨损。图 4 中黏附力的变化来源于醇分子在滑动界面的吸附。摩擦力常被建模为剪切应力乘以接触面积,而 Koren 等^[30-31]的研究表明,接触面积可以用含有黏附力的一系列参数进行数学建模,表明摩擦力的大小会受到黏附力的影响。因此,不同的黏附力一定程度上会导致不同样品表面测得摩擦力的差异,考虑到石墨表面摩擦力信号随醇的变化跨度较大,而黏附力的变化相对较小,下面从微观层面分析该摩擦行为。

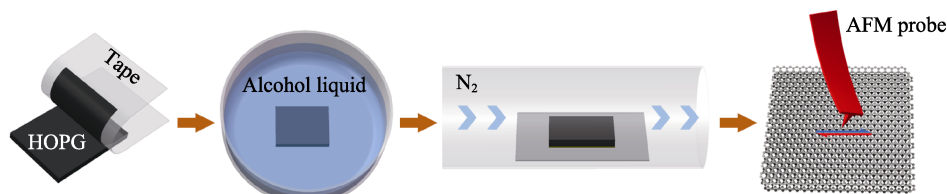


图 1 石墨样品处理的实验示意图

Fig.1 Experimental diagram of graphite sample preparation

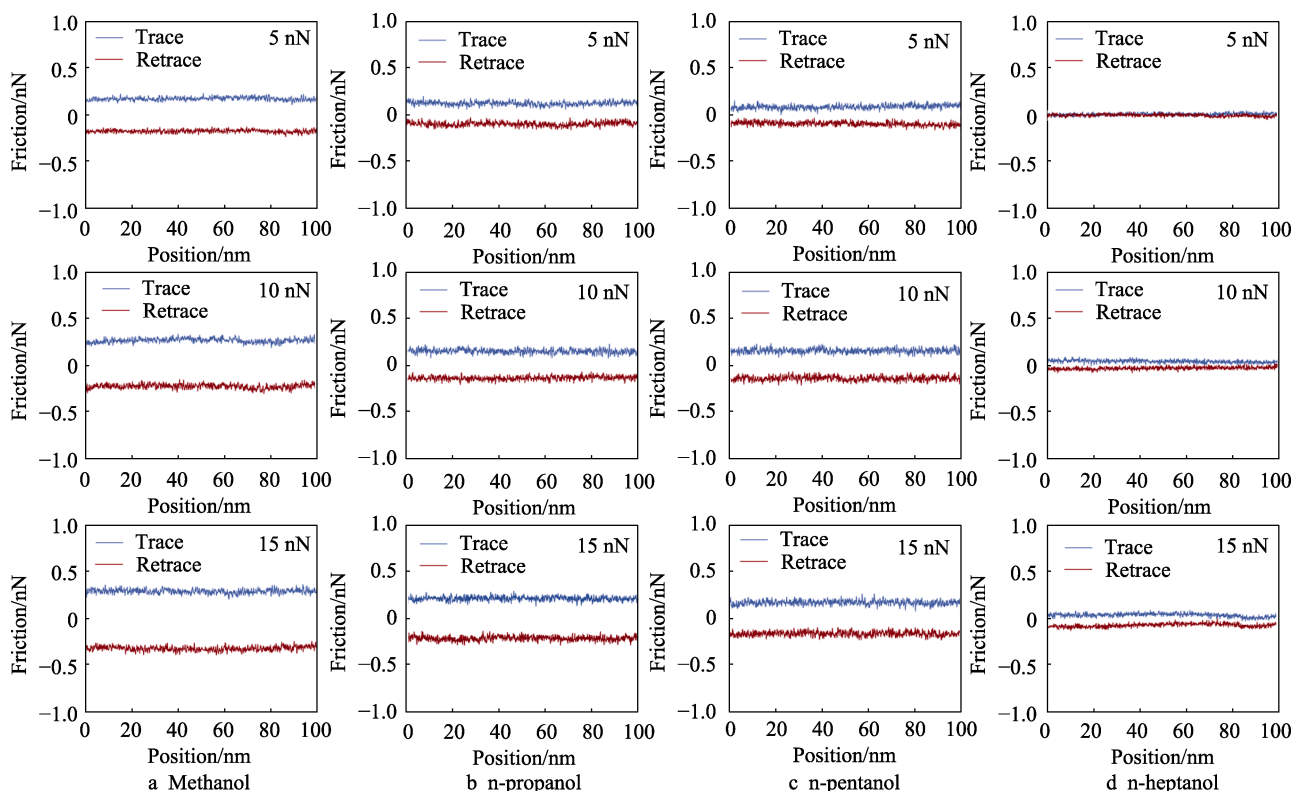


图2 AFM 探针在不同醇中处理过的石墨表面测得的摩擦力信号

Fig.2 Friction signal for the sliding of an AFM tip over the graphite surfaces treated with different alcohols

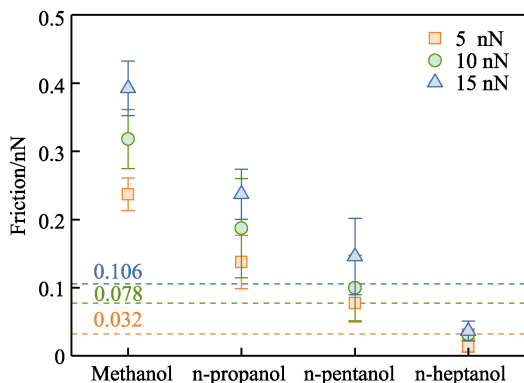


图3 在不同醇中处理过的石墨表面的平均摩擦力
(图中虚线代表未处理石墨表面的平均摩擦力水平)
Fig.3 Average friction forces on the graphite surfaces treated with different alcohols (dash lines represent average friction forces on untreated graphite surfaces)

在醇中取出并在氮气中吹干的石墨表面吸附有醇分子,而二氧化硅表面通常嫁接有羟基和醚键,已有研究表明醇分子容易通过氢键物理吸附在 SiO_2 表面^[32-33]。与之类似的,在滑动过程中残留在石墨表面的醇分子会吸附在 AFM 探针的 SiO_2 针尖上,这些吸附分子在接触界面上起到了润滑作用。不同醇分子在润滑能力上的差异来源于它们不同的分子构象熵^[25],醇分子的分子构象熵随着碳链的增长而增加,具有较大分子构象熵的正庚醇分子吸附在接触界面,增强了针尖与石墨接触的非公度性,促进了界面滑动,使得石墨表面的摩擦力更低。而碳链较短的醇分子(如乙

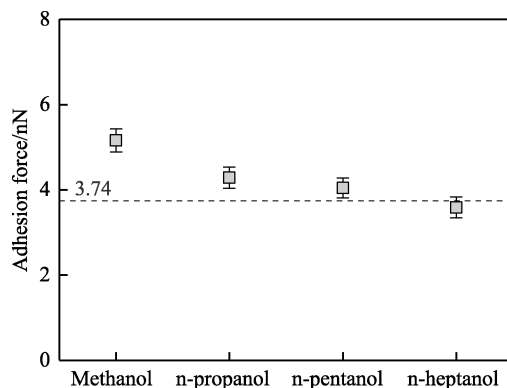


图4 在不同醇中处理过的石墨表面的黏附力
(图中虚线代表未处理石墨表面的黏附力水平)
Fig.4 Adhesion forces measured on the graphite surfaces treated with different alcohols (dash lines represent adhesion force measured on untreated graphite surfaces)

醇)分子构象熵较小,反而阻碍了滑动。

另一方面,摩擦往往伴随着热量的产生。由 AFM 探针针尖上的微凸体与石墨烯接触形成的实际接触面积远小于名义接触面积,使得摩擦副接触点附近温升比摩擦表面平均温度的温升高许多倍,促进了醇分子的分解和重组,使摩擦化学反应更容易发生^[34]。Hibi 等^[35-36]的研究表明, SiO_2 中的 Si 原子容易受到醇氧原子的攻击而发生亲核取代反应,形成硅酸盐 ($\text{Si}-\text{OR}$, 其中 R 代表烷基),生成的硅醇盐在水解和缩合反应下又进一步转化为硅胶。这些硅胶的存在增大了针尖与石墨之间的接触面积,从而导致摩擦力

和黏附力的增大。此外, 随着醇分子尺寸的增大, 亲核性降低, 而低碳醇分子在亲核取代过程中更容易接近硅原子, 摩擦化学反应速率更快^[37], 因此低碳醇中处理过的石墨表面具有更大的摩擦力和黏附力。

3 结论

通过纳米尺度摩擦试验, 研究了醇分子对石墨基面上摩擦行为的影响, 其可作为调节石墨摩擦学性能的一种方法。结果表明, 接触表面的公度性和摩擦化学反应产物共同影响了摩擦副表面的接触情况, 从而影响了 SiO_2 针尖在石墨表面的摩擦学行为。得出了以下结论:

1) 在不同醇中浸泡过后石墨表面的摩擦学性能会发生显著改变。醇分子的碳链越长, 处理后的石墨表面摩擦力越小, 且黏附力也随之减小。

2) 吸附在石墨表面的醇分子会通过改变接触公度性来影响摩擦力。醇分子的碳链越长, 其分子构相熵越大, 降低了接触公度性, 从而减小摩擦力。

3) 接触界面的摩擦化学反应产物不利于表面润滑, 而碳链越长的醇分子亲核性越低, 反应速率越慢, 因此受到摩擦化学反应的影响就越小。

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