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二聚酸离子液体作为水基润滑添加剂的 摩擦学性能研究

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摘要: 以不同链长的二元脂肪酸作为阴离子, 以二异丙醇胺作为阳离子, 合成了4种羧酸铵型离子液体(CAILs), 并研究了其作为水-二乙二醇(WDG)添加剂的溶解性和摩擦学性能, 随后采用光学显微镜(OM)、扫描电子显微镜(SEM)、聚焦离子束-透射电子显微镜(FIB-TEM)、石英晶体微天平(QCM)和X射线光电子能谱(XPS)对试验后的磨痕进行二维形貌的观察和化学组成的分析. 结果表明: 4种离子液体在水-二乙二醇中有良好的溶解性(>4.0%, 质量分数)和稳定性. 此外, 4种离子液体都能增强WDG的减摩性能, 但只有二聚酸离子液体(DC₃₆-DIPA)增强了WDG的抗磨性能, 同时提高了WDG的承载能力(500 N), 表现出最佳的摩擦学性能. OM、SEM、FIB-TEM、QCM和XPS结果表明: 质量分数0.5% DC₃₆-DIPA在钢-钢摩擦副表面生成了厚度约为60~70 nm、以铁氧化物为主要成分的摩擦膜, 这层摩擦膜通过DC₃₆-DIPA分子在金属表面的吸附以及润滑剂与摩擦副之间的摩擦化学反应而形成, 起到减摩抗磨的作用.

关键词: 离子液体; 吸附; 摩擦化学反应; 润滑机制

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Tribological Properties of Dimer Acid Ionic Liquids as Lubricant Additives in Water-Based Fluid

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Abstract: In this study, four ammonium carboxylate-type ionic liquids (CAILs) were synthesized with four dicarboxylic acids and diisopropanolamine and used as water-based lubricant additives, with their tribological performance and lubrication mechanisms investigated. Infrared spectroscopy and nuclear magnetic resonance (NMR) were used to show

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that the synthesized CAILs additives had well-defined molecular structures. The four CAILs had been shown to have good solubility and dispersion stability in water-diethylene glycol (WDG) solution. Tribological tests of the four CAILs in WDG were carried out using a TE77 long-range reciprocating friction tester to evaluate the lubrication performance of them as additives in a ball-and-disc configuration for steel-steel sliding contacts. The four CAILs reduced the friction coefficients of the WDG base fluid to varying extents but only the dimeric acid ionic liquids (DC₃₆-DIPA) effectively reduced the wear volume, suggesting that DC₃₆-DIPA possessed the best friction reduction and anti-wear performance under the studied conditions. The influence of concentration of DC₃₆-DIPA was further examined at four concentrations of 0.5%, 1.0%, 1.5%, and 2.0% (mass fraction). Results showed that the friction coefficients did not show any significant difference, a concentration of 0.5% DC₃₆-DIPA exhibited the smallest wear volume. This suggested that the concentration could significantly affect the anti-wear performance of the water-based lubricants. It may be related to the change of molecular structure and existing status of DC₃₆-DIPA in the base fluid with effects such as self-assembly. To verify this speculation, small angle scattering (SAXS) tests were performed. It was found that DC₃₆-DIPA exhibited scattering differences at different concentrations, which may indicate that DC₃₆-DIPA exists in different molecular morphology in the solution. It may undergo a transition from vesicle to micelle for the four concentrations. This could be the main reason for the large difference in the anti-wear performance of DC₃₆-DIPA at different concentrations. In addition, the DC₃₆-DIPA additive in the WDG could with stand loads of at least 500 N, and it exhibited low and stable friction coefficients in the 50~500 N range. Optical microscopy and scanning electron microscopy were used to analyze the wear surfaces lubricated with the WDG base fluid and 0.5% DC₃₆-DIPA. Compared to the base fluid, the wear track produced by 0.5% DC₃₆-DIPA was narrower, more uniform and more smooth. QCM tests demonstrated that the WDG base fluid produced obvious physical adsorption, and the addition of DC₃₆-DIPA in WDG had negligible effect on the adsorption. In addition, the XPS analysis results also showed that a generation of tribo-film with iron oxides as the main component in the presence of DC₃₆-DIPA. FIB-TEM results showed that the thickness of the film was 60~70 nm. In conclusion, DC₃₆-DIPA had excellent friction reduction and load-carrying capacity performances, which could be mainly attributed to the physical/chemical adsorption of polar groups -COO⁻ in DC₃₆-DIPA. The excellent anti-wear performance was due to the tribo-chemical reaction between DC₃₆-DIPA molecules and the steel disc, which generated a tribo-film of rather uniform thickness. This study provided an example as a reference for the research and application of ionic liquids in the field of water lubrication.

Key words: ionic liquids; adsorption; tribo-chemical reaction; tribological mechanism

摩擦磨损造成的机械设备损耗是能源浪费的主要原因之一,合理使用润滑剂作为工作介质可以有效减少摩擦磨损延长机械设备的使用寿命,减少能源的损耗,提高能源的利用率^[1-3]。目前的润滑剂可以分为固体材料、半固体材料和流体材料3种。其中,液体润滑材料作为流体材料的1种,其在实际应用中更为广泛。在液体润滑材料中,矿物油等油基润滑产品润滑性能优良,能够有效减少机械设备的摩擦磨损,增加设备的使用寿命,因此被广泛应用于工业领域,例如发动机油、齿轮油和液压油等。但油基润滑产品也有低闪点和易燃烧等缺点,这就导致它们在某些高温和易爆炸等工况下的应用受到限制;较低的生物降解性使其容易在排放后污染生态环境,不符合国家对于绿色环保的要求^[4-5]。因此,开发高性能且绿色无污染的新型润滑剂来代替油基润滑产品至关重要。

水是1种成本低且环保无污染的材料,相比矿物油等油基材料而言,其优异的清洗、冷却以及难燃的特性引起人们越来越广泛的关注,良好的抗燃性让水

基润滑材料在难燃液压液领域有着巨大的应用价值。但较低的黏度使得水难以在机械零部件之间形成有效的润滑保护膜,导致润滑效果不佳^[6-9]。此外,水会导致金属材料的腐蚀或锈蚀。为了解决这一问题,向水中加入润滑剂、防锈剂以及腐蚀抑制剂等功能性添加剂是增强水基润滑剂性能的有效手段之一,也是水基金属加工液和水基难燃液液压等水基润滑产品的研究和应用过程中必不可少的环节^[10-13]。

离子液体(ionic liquids, ILs)是由有机阴阳离子组成的1种有机熔融盐,其独特的物理特性和化学特性使其在润滑材料领域得到广泛关注。自2001年刘维民团队首次报道了离子液体应用于润滑领域的研究以来,研究人员对其用作纯润滑剂以及润滑添加剂的可能性进行了大量的研究,离子液体已被证实可有效改善抗磨性能和防腐蚀性能而应用于水基润滑材料当中^[14-16]。Phillips等^[17]首次开展了将离子液体用作水润滑添加剂的研究,其结果表明:在流体润滑状态下,向水中添加质量分数2.0%的[C₄C₁im]BF₆/PF₆后,可有

效缩短氮化硅陶瓷的磨合周期, 在较短的时间内就表现出稳定且较低的摩擦系数. Wang等^[18]设计制备了2种布洛芬基离子液体, 并通过试验证明了其在水-乙二醇中有良好的润滑性能和抗腐蚀性能. Zheng等^[19]设计制备了2种质子型离子液体, 通过研究发现其在一定复配比例的条件下能够显著增强水-甘油溶液的润滑性能. 离子液体不仅本身具有良好的特性, 而且能与部分物质起到协同润滑的作用, 例如生物材料^[20]、无机化合物^[21]以及碳量子点^[22]等. 虽然离子液体已经得到了诸多研究, 但仍然存在合成过程复杂^[23]、易造成腐蚀^[24]、高温或低压下的性能不佳^[25]等问题. 因此, 开发易制备且环境友好性较强的水基润滑添加剂至关重要.

二聚酸是一种含有2个羧基的不饱和脂肪酸, 通常由油酸、亚油酸等不饱和的脂肪酸聚合而成, 目前已经广泛应用于涂料、表面活性剂、润滑剂和油墨印刷等领域, 是一种重要的油脂化工产品^[26-27]. 由于具有极性的羧基和2条较长的非极性碳链, 所以二聚酸分子能够通过吸附在活性金属表面形成较为稳定的摩擦保护膜, 减少金属材料的摩擦和磨损. 早在2001年, 周灿丰等^[28]就使用具有单环结构的二聚酸和氢氧化钾合成了二聚酸钾, 并将其添加于水-乙二醇液压基础液中, 用四球摩擦磨损试验机进行了摩擦学测试. 结果表明, 与进口产品和水溶性改性二烷基二硫代磷酸锌(ZDDP)相比, 二聚酸钾的添加能够更有效地减小四球磨斑直径, 抗磨性能明显增强. 张文田等^[29]制备了二聚酸-二乙醇胺盐, 通过研究发现, 仅质量分数为1.0%

的二聚酸-二乙醇胺盐就能显著增强水-乙二醇基础液的摩擦学性能, 特别是 P_B 由147 N提高到981 N, 极大地提高了基础液的承载能力. 可见, 二元酸衍生物作为水溶性抗磨添加剂具有较好的抗磨损性能.

基于水-乙二醇难燃液液压对抗磨润滑添加剂的需求, 我们利用脂肪酸中的羧基和醇胺中氮原子之间能够发生质子转移这一特性, 设计制备了4种包括二聚酸离子液体在内的羧酸铵型离子液体(CAILs), 并将其用于水-乙二醇的添加剂. 通过摩擦学测试和对磨损表面的表征, 对比了二聚酸离子液体(DC₃₆-DIPA)和其他离子液体的摩擦学性能, 并探究分子结构、浓度、载荷和频率对二聚酸离子液体摩擦学性能的影响, 旨在揭示二聚酸离子液体可能的润滑机理, 可为二聚酸及其衍生物在润滑领域的应用提供一定参考.

1 试验部分

1.1 离子液体的制备

本研究中所制备出的离子液体结构如图1所示, 合成所用的二元酸和醇胺以及其他清洗所用的试剂等均来自国药集团化学试剂有限公司, 纯度均为分析纯且不需要进一步纯化.

离子液体制备的具体步骤^[30]如下: 首先向三口烧瓶中加入二异丙醇胺(26.6 g, 0.2 mol); 在氮气保护和磁力搅拌下, 加入癸二酸(20.2 g, 0.1 mol), 随后升温至80 °C, 持续搅拌反应8 h, 得到癸二酸-二异丙醇胺离子液体(DC₁₀-DIPA). 使二元酸和二异丙醇胺保持

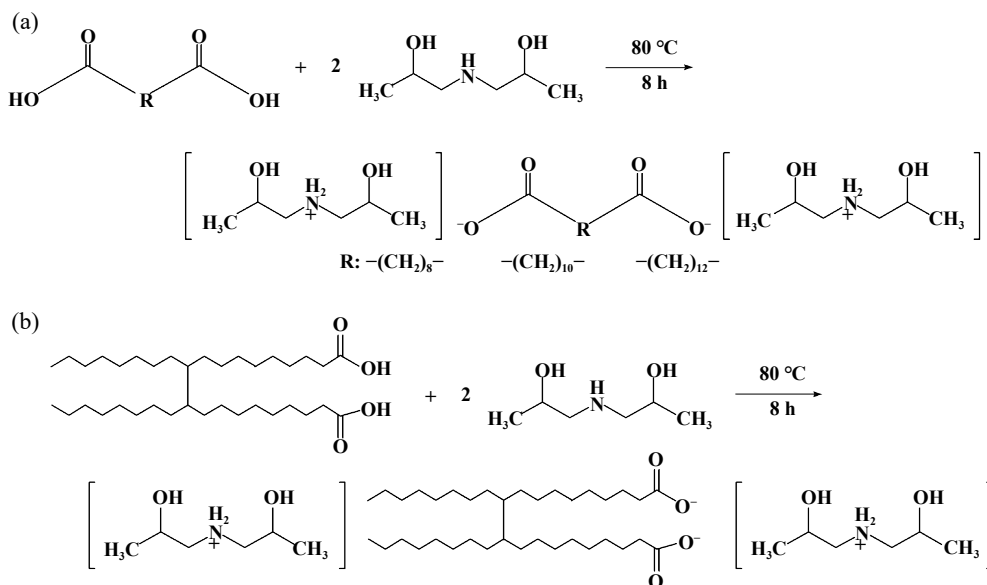


Fig. 1 synthetic route of ILs: (a) DC₁₀-DIPA, DC₁₂-DIPA and DC₁₄-DIPA; (b) DC₃₆-DIPA

图1 离子液体的制备路线: (a) DC₁₀-DIPA, DC₁₂-DIPA和DC₁₄-DIPA; (b) DC₃₆-DIPA

1:2的摩尔比,采用同样的步骤分别制备出十二烷二酸-二异丙醇胺离子液体(DC₁₂-DIPA)、十四烷二酸-二异丙醇胺离子液体(DC₁₄-DIPA)和二聚酸-二异丙醇胺离子液体(DC₃₆-DIPA).在常温下,4种离子液体均为液体.基础液由去离子水和二乙二醇按1:1的质量比配制而成,记为WDG.

采用傅里叶红外光谱仪(FIR)对不同离子液体的分子结构做了初步表征,结果如图2所示.可以看出,位于1 650~1 750 cm⁻¹处属于-COOH的特征峰被削弱,而位于1 550~1 570 cm⁻¹处属于-COO⁻的非对称特征峰以及位于1 395~1 460 cm⁻¹处属于-COO⁻的对称特征峰出现,证明了离子液体的成功合成^[31].

选用4种二元酸离子液体中的DC₁₀-DIPA,采用核磁共振对其分子结构作进一步表征,结果如图3所示.在DC₁₀-DIPA核磁共振氢谱中,代表-COOH化学位移的特征峰(11~13 ppm)消失,但是代表-NH₂⁺的特征峰(4.82 ppm)出现,因此可以说明-COOH中的氢已经转移到了氨基中,从而进一步证明了离子液体的成功合成.

1.2 水溶性与黏度

将一定质量的离子液体溶解在水-二乙二醇溶液中,制备质量分数为4.0%的混合溶液,混匀后静置,分别于静置30 min和30 d后观察其外观.采用斯塔宾格

全自动运动黏度测试仪(Anton Paar GMBH, Austria)测试不同润滑剂在25和40 °C下的运动黏度.

1.3 摩擦学测试

采用TE77长程高频往复摩擦试验机(Phoenix Instrumentation, UK)评价离子液体作为水基润滑添加剂在钢-钢摩擦副上的摩擦学性能.试验采用球-盘点接触,上试球的规格为100Cr6,直径10 mm,洛氏硬度(HRC)为62.8±1.0,粗糙度(R_a)为0.020 μm,下试盘的规格为洛氏硬度64±1.0,粗糙度为0.020 μm.试验条件温度为30 °C,法向载荷为50 N,往复运动频率为10 Hz,往复运动距离为11 mm,试验时间为30 min.在变载荷试验中,载荷以50 N/3 min的速度变化,变化范围为50~500 N;在变频试验中,频率以2.5 Hz/5 min的速度变化,变化范围为2.5~15 Hz.将上试球和下试盘固定好后,开启程序,对不同的试样进行摩擦磨损测试.摩擦磨损测试结束后,依次用无水乙醇和石油醚清洗下试盘,采用划痕仪(Rtec instrument, America)获取试验后的磨损体积,根据得到的摩擦系数和磨损体积,评价离子液体在水-二乙二醇中的摩擦学性能.

1.4 磨损测试

摩擦试验结束后,采用光学显微镜(OM, Spectro Scientific AMETEK, America)和扫描电子显微镜(SEM,

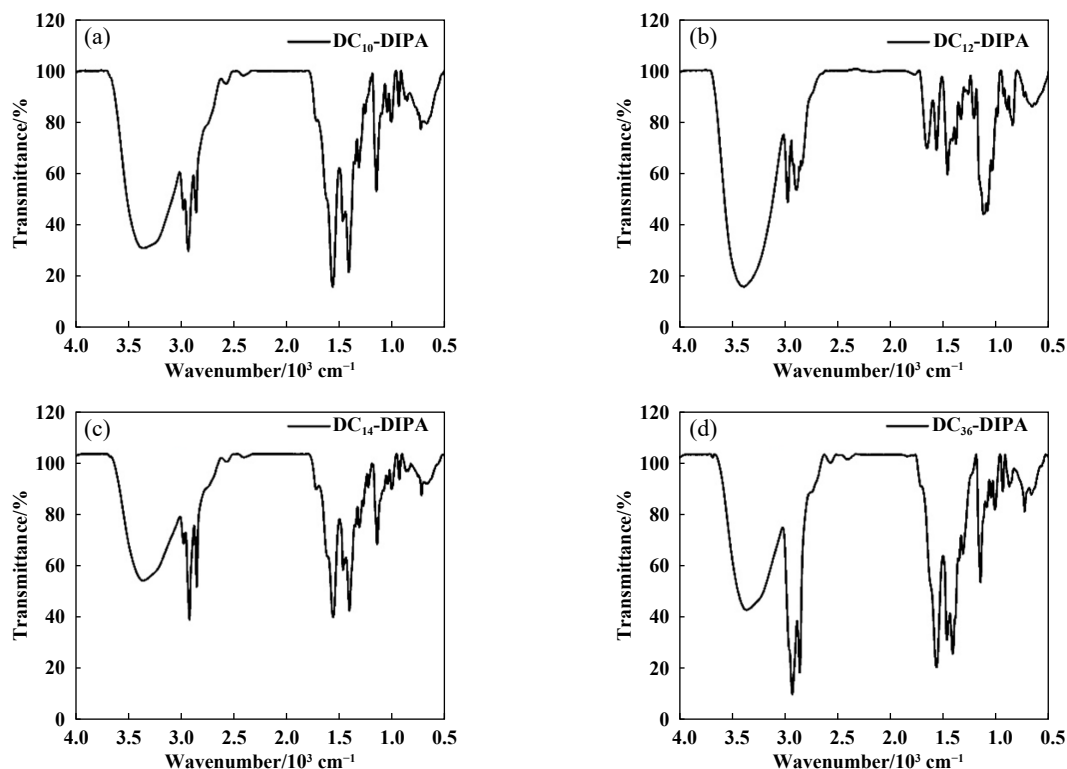
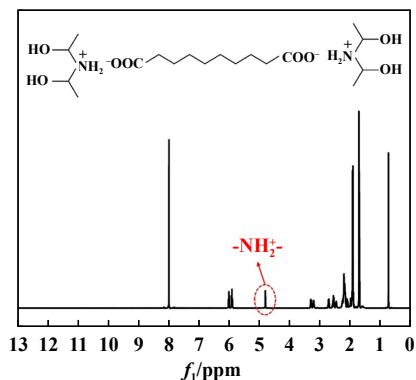


Fig. 2 FTIR spectra of ILs: (a) DC₁₀-DIPA; (b) DC₁₂-DIPA; (c) DC₁₄-DIPA; (d) DC₃₆-DIPA

图2 离子液体的红外光谱图:(a) DC₁₀-DIPA; (b) DC₁₂-DIPA; (c) DC₁₄-DIPA; (d) DC₃₆-DIPA

Fig. 3 ^1H NMR spectrum of DC₁₀-DIPA图 3 DC₁₀-DIPA的核磁共振氢谱图

Thermo Fisher Scientific, America)获取摩擦试块表面磨损区域的二维形貌。

采用共聚焦离子束(FIB, Thermo Fisher Scientific, America)对磨损痕迹的特定部位进行切割,采用透射电镜(TEM, Thermo Fisher Scientific, America)获取切割部位横截面的二维形貌,观察摩擦膜的厚度和形态,同时采用EDS检测元素组成。

在Thermo Scientific™ K-Alpha™+光谱仪(Thermo Fisher Scientific, America)采用X射线光电子能谱(XPS)分析磨损表面特定元素的化学状态,该设备用Al-K α 作为射线源,样品在真空中($P < 10^{-8}$ mbar)进行检测。用Casaxps软件对试验峰进行拟合。所有的峰都用污染的C 1s峰结合能284.8 eV进行校准。

1.5 X射线小角度散射(SAXS)试验

为了探究离子液体在基础液WDG中的存在形态,进行了小角度散射(SAXS)试验。本研究中的SAXS试验采用了法国的X射线小角度散射仪,型号为Xeuss 2.0,试验中所使用铜靶光管功率为30 W,波长为1.541 89 Å,探测器为Pilatus 3R 300K,相机单个像素大小为172 μm 。将少量样品置于样品池中,在25 °C下用X射线照射10 min,用每个样品的测量结果扣除背景(WDG)的测量结果,最后得到散射曲线,根据散射曲线判断离子液体在基础液中的形态。

1.6 QCM测试

在25 °C的条件下,采用蠕动泵将试样经聚四氟乙烯管通入含镀金石英晶体传感器的腔室中。试验过程中,液体流速设为50 $\mu\text{L}/\text{min}$,传感器采用镀金石英晶体(QSX-301, Q-sense AB)。试验时,先通入去离子水,直至基线稳定,随后通入含离子液体的WDG溶液,稳定一段时间后,观察频率变化。使用Q-soft 401软件记录不同基频(3, 5, 7, 9, 11, 13)上频率的变化值(Δf),选择基频为3时的 Δf 进行分析和讨论。

2 结果与讨论

2.1 水溶性与黏度

将本试验中制备的离子液体以质量分数4.0%溶解在水-二乙二醇溶液中,混合溶液的外观如图4所示。可以看到,添加离子液体前后溶液的外观没有明显的差异,说明在室温下,所制备的离子液体在水-二乙二醇中具有良好的溶解性。此外,静置30 d后,溶液外观保持清澈透明,表明离子液体在水-二乙二醇中具有良好的稳定性。

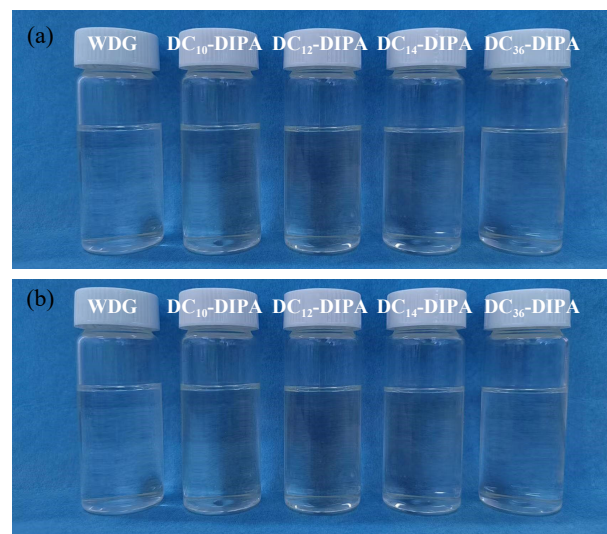


Fig. 4 Appearance of WDG containing ILs at a mass fraction of 4.0%: (a) mix evenly and stand for 30 minutes; (b) mix evenly and stand for 30 days

图 4 4.0%离子液体的水溶性: (a)混匀后静置30 min; (b)混匀后静置30 d

黏度是润滑剂重要的参数之一,黏度的大小反映了流体内部的分子相互作用,适当的黏度有利于润滑剂在润滑界面上形成强度适宜的摩擦膜,从而获得最佳的摩擦学性能^[32]。不同润滑剂在25 °C和40 °C下的运动黏度列于表1中(其中润滑剂添加剂均为质量分数,全文同)。由表1可知,4种含离子液体的润滑剂在25和40 °C下的运动黏度范围分别为3.096~3.279和2.071~2.164 mm^2/s ,而WDG在25 °C和40 °C的运动黏度分别为3.004和2.036 mm^2/s ,离子液体对WDG的黏度增加幅度不超过6%,说明离子液体对WDG的黏度影响较小,因此对润滑膜的组成和结构影响也较小。

2.2 摩擦学性能

2.2.1 分子结构的影响

图5所示为4种羧酸铵离子液体的摩擦系数曲线和磨损体积。可以看出,不同离子液体添加剂对于基础液的摩擦学性能有着不同的影响。从图5(a)中可以看出,WDG在开始最初的50 s内摩擦系数迅速升高至

表 1 不同润滑剂的在25和40 °C下的运动黏度
Table 1 Kinematic viscosity of different lubricants at 25 and 40 °C

| Aqueous lubricants | Kinematic viscosity/(mm ² /s) | |
|-----------------------------|--|-------|
| | 25 °C | 40 °C |
| WDG | 3.004 | 2.036 |
| 0.5% DC ₁₀ -DIPA | 3.134 | 2.071 |
| 0.5% DC ₁₂ -DIPA | 3.096 | 2.096 |
| 0.5% DC ₁₄ -DIPA | 3.234 | 2.127 |
| 0.5% DC ₃₆ -DIPA | 3.279 | 2.164 |

0.46, 然后迅速下降, 经500 s的磨合后稳定在0.13~0.14. DC₁₀-DIPA经200 s磨合后稳定在0.105上下, 但从600 s开始摩擦系数出现了多个持续时间极短但较高的峰值; DC₁₂-DIPA经300 s磨合后稳定, 从600 s开始到结束, 摩擦系数水平与WDG相当; DC₁₄-DIPA仅在200~530 s内有短暂的波动, 在这之外的时间内摩擦系数稳定在0.09~0.10; DC₃₆-DIPA磨合时间极短, 整个过程都稳定在0.08上下. 在相同条件下, 二聚酸离子液体(DC₃₆-DIPA)具有最佳的减摩性能, 这表明长碳链在摩擦过程中有助于减少水平方向上的剪切应力, 在表面形成了吸附膜, 减少了摩擦. 从图5(b)中可以看出, 磨损体积大小顺序为DC₁₀-DIPA≈DC₁₂-DIPA>DC₁₄-DIPA>WDG>DC₃₆-DIPA, 只有DC₃₆-DIPA改善了WDG的抗磨性能, 而其他离子液体添加剂均导致WDG的抗磨性能的下降. 可见, DC₃₆-DIPA在水-二乙二醇中的减摩抗磨性能最佳, 这可能是由于较长的阴离子烷基链增加了分子的柔性, 同时DC₃₆-DIPA中独有的支链结构使其稳定性更好, 因此生成的摩擦膜更牢固, 能够有效减少摩擦副之间的直接接触, 从而显著降低摩擦和磨损^[31].

2.2.2 添加浓度的影响

图6所示为不同浓度的DC₃₆-DIPA的摩擦系数曲线和磨损体积. 从图6(a)中可以看出, DC₃₆-DIPA在质量分数为0.5%和1.0%下的摩擦系数最低且最为稳定,

二者水平相近, 而质量分数为1.5%和2.0%摩擦系数相对较高且不稳定. 而从图6(b)可以看出, 磨损体积大小顺序为1.5% DC₃₆-DIPA>2.0% DC₃₆-DIPA>1.0% DC₃₆-DIPA>0.5% DC₃₆-DIPA, 0.5% DC₃₆-DIPA磨损体积最小, 为 $5.269 \times 10^{-4} \text{ mm}^3$, 说明DC₃₆-DIPA在质量分数为0.5%下的抗磨性能最佳. Gu等^[34]研究发现, 2-己基癸酸钠添加到水中时, 其摩擦学性能随浓度的变化而变化, 较低添加浓度会形成囊泡, 摩擦磨损较低, 而较高浓度则形成胶束, 减摩抗磨性能变差. DC₃₆-DIPA在质量分数为0.5%下可能已经形成囊泡, 囊泡的存在使其在开始的极短时间内就生成了连续且相对稳定的摩擦膜, 这层摩擦膜能够有效降低摩擦过程中的剪切应力, 在减少摩擦和磨损方面起关键作用. 而在DC₃₆-DIPA其他浓度下可能已经形成胶束, 因此抗磨损性能不如质量分数为0.5% DC₃₆-DIPA.

2.2.3 载荷和频率的影响

图7(a-b)所示分别为0.5% DC₃₆-DIPA在变载和变频条件下的摩擦系数. 在图7(a)中, WDG在整个过程中出现了多个较高的峰值, 且波动幅度较大, 摩擦系数均高于0.125, 当载荷增加到400 N后, 由于摩擦力超过设定上限, 试验被迫停止, 这说明WDG在变载条件下的润滑性能较差. 0.5% DC₃₆-DIPA在变载的整个过程中表现出比WDG低的摩擦系数, 特别是当负载增加到100 N后, 摩擦系数比WDG减少了40%以上, 能够稳定在0.075左右. 同样地, 在图7(b)中, WDG波动幅度依然较大, 摩擦系数在0.15以上, 而0.5% DC₃₆-DIPA的摩擦系数稳定在0.12~0.13. 可见, 0.5% DC₃₆-DIPA在高负载和高频率下依然具有优良的减摩性能, 这可能是因为在较低的载荷或频率下, 离子液体润滑剂在润滑界面上已经形成了较为稳定的吸附膜与化学反应膜, 因此即使在高负载和高频率下, 其摩擦系数依然较低.

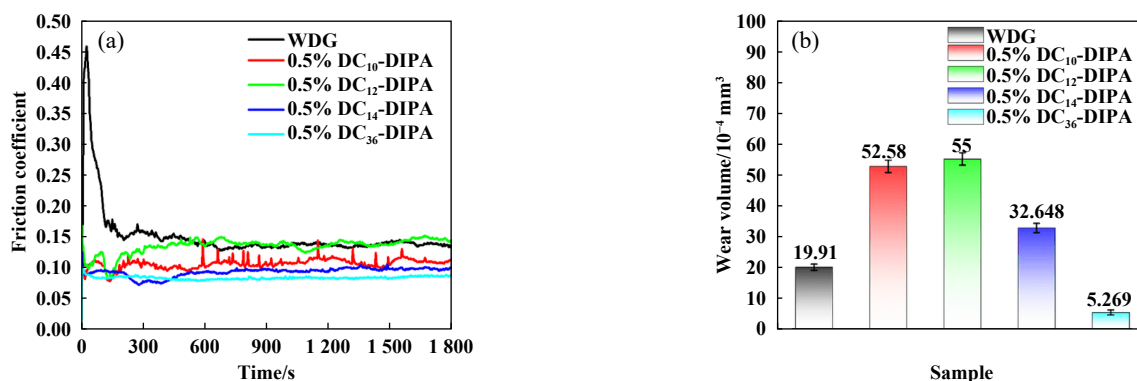


Fig. 5 (a) Friction coefficients and (b) wear volumes of ammonium carboxylate ILS

图 5 不同羧酸铵离子液体的(a)摩擦系数和(b)磨损体积

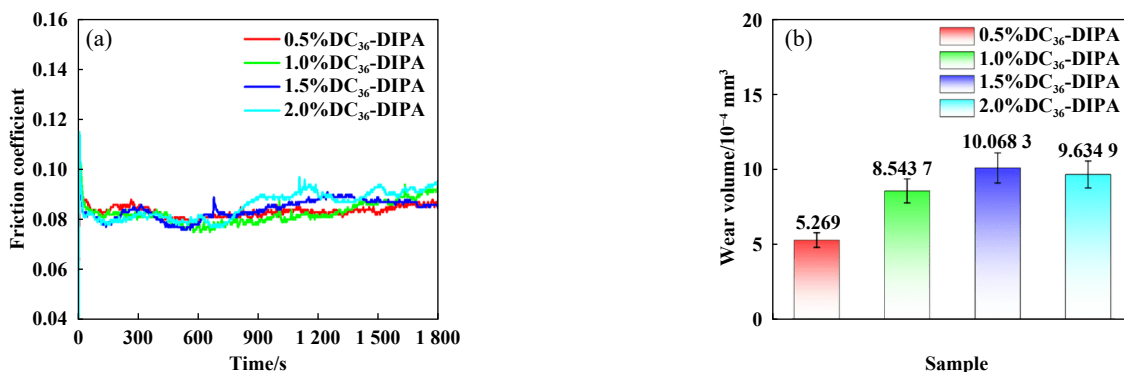


Fig. 6 (a) Friction coefficients and (b) wear volumes of DC₃₆-DIPA at different concentrations in WDG

图 6 不同浓度的DC₃₆-DIPA的(a)摩擦系数和(b)磨损体积

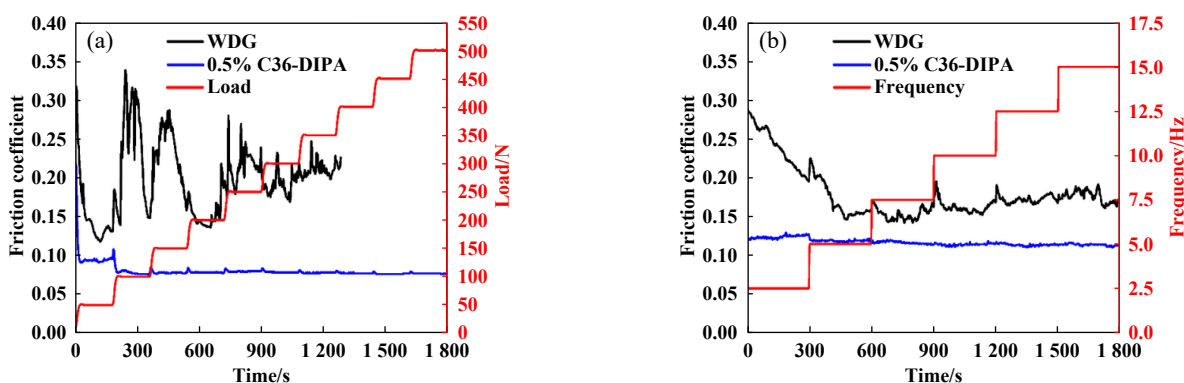


Fig. 7 Friction coefficient of 0.5% DC₃₆-DIPA under (a) load conversion and (b) frequency conversion

图 7 0.5% DC₃₆-DIPA在(a)变载和(b)变频条件下的摩擦系数

2.3 磨损表面分析

为了更直观地了解离子液体对水-二乙二醇体系抗磨性能的影响,采用光学显微镜对磨损区域进行了观察.图8所示为WDG、0.5% DC₁₄-DIPA和0.5% DC₃₆-DIPA润滑条件下,磨损痕迹在光学显微镜下的照片.可以看到,WDG润滑后产生的磨痕较宽,表面出现了磨粒磨损,磨损痕迹两端的边缘处有类似磨屑的物质残留.0.5% DC₁₄-DIPA磨痕比WDG颜色更深,对应磨损体积增加,磨痕周围还有大量磨损后的残留物质.而0.5% DC₃₆-DIPA润滑后产生的磨痕更窄,表面更均一,这表明0.5% DC₃₆-DIPA可能在金属表面形成了有效的摩擦保护膜.

图9(a~c)所示分别为WDG、0.5% DC₁₄-DIPA和0.5% DC₃₆-DIPA润滑剂的SEM照片.可以看出,WDG产生的磨痕较宽,表面存在数量较多且较为密集的凹坑,并且有多处轻微的剥落,说明在摩擦过程中,WDG中的水在摩擦副表面产生了较为严重的磨粒磨损,润滑性能不佳;0.5% DC₁₄-DIPA润滑后产生的磨痕表面有大量的犁沟,说明存在严重的磨粒磨损;0.5% DC₃₆-

DIPA润滑后产生的磨痕表面磨损形式主要为黏着磨损,宽度明显比WDG和0.5% DC₁₄-DIPA更窄. SEM结果与摩擦学试验结果相一致,表明0.5% DC₃₆-DIPA能够在润滑界面上形成摩擦保护膜,显著减少了摩擦磨损.

2.4 摩擦膜分析

为了观察摩擦膜的形貌,探究摩擦膜的化学组成,对0.5% DC₃₆-DIPA润滑后的磨损痕迹进行FIB切割,进而获取横截面的透射电镜照片和EDS元素分布,结果如图10所示.可以看出,0.5% DC₃₆-DIPA经摩擦测试后在金属表面形成了厚度较为均一的摩擦膜,摩擦膜厚度在60~70 nm左右.此外,在图10(d)中,主要元素随深度的变化呈梯度分布,特别是O和Fe,根据元素强度可以判断,5~65 nm处为摩擦膜的区域,且摩擦膜主要由铁氧化物组成,这些氧化物可能来自润滑剂和摩擦副的化学反应. FIB-TEM的结果有力地证明了0.5% DC₃₆-DIPA在摩擦界面上形成了强度适宜的摩擦保护膜,正是这层膜的存在有效减少了摩擦和磨损.

2.5 SAXS分析

为了验证2.2.2中对于DC₃₆-DIPA在基础液WDG

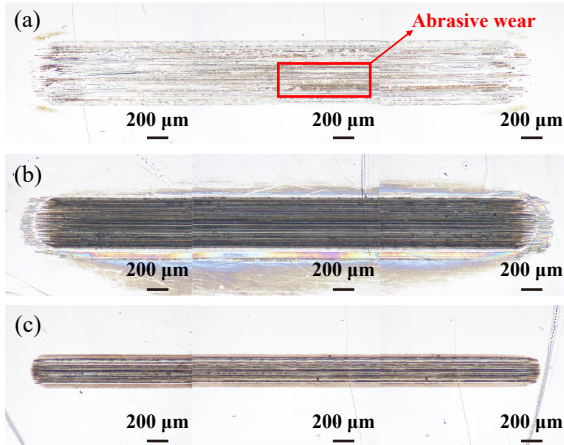


Fig. 8 Optical micrographs of worn area lubricated by different lubricants: (a) WDG; (b) 0.5% DC₁₄-DIPA; (c) 0.5% DC₃₆-DIPA

图 8 不同润滑剂润滑后表面的光学显微镜照片:
(a) WDG; (b) 0.5% DC₁₄-DIPA; (c) 0.5% DC₃₆-DIPA

中存在形态的推测, 对不同浓度的DC₃₆-DIPA进行了 SAXS测试, 结果如图11所示. 通过对低散射矢量区域的曲线进行线性拟合, 发现拟合后曲线的变化率随浓度增加呈现出先增加后减小的变化情况, 这意味着

DC₃₆-DIPA在基础液WDG中可能经历了3种形态的转变, 因而散射强度受到影响而发生变化. 具体来说, DC₃₆-DIPA在质量分数为0.5%时, 在基础液中以囊泡的形式存在; 而在质量分数为1.0%和1.5%时已经开始向胶束转变, 溶液中既有囊泡, 也存在胶束; 在质量分数为2.0%下, 其变化率减小, 表明囊泡完全转变为胶束^[35]. 结合2.2.2的试验结果, 说明DC₃₆-DIPA在质量分数为0.5%下在基础液WDG中以囊泡的形态存在, 囊泡对于抗磨性能起关键作用, 而胶束的存在会使得抗磨性能变差.

2.6 QCM分析

为了测试离子液体添加剂在金属表面的吸附能力, 采用镀金的石英晶体芯片对含0.5% DC₃₆-DIPA和1.5% DC₃₆-DIPA的WDG溶液进行了试验, 结果如图12所示. 可以看出, 0.5% DC₃₆-DIPA的加入使得基础液WDG频率变化(Δf)由500 Hz增加到520 Hz, 1.5% DC₃₆-DIPA使 Δf 增加到530 Hz, 用水冲洗后频率均恢复到初始水平. QCM结果表明, 在整个试验过程中, 所有溶液在镀金芯片表面均产生了物理吸附, 虽然DC₃₆-DIPA浓度的增加会增加 Δf , 但相比WDG来说变化较小. 另

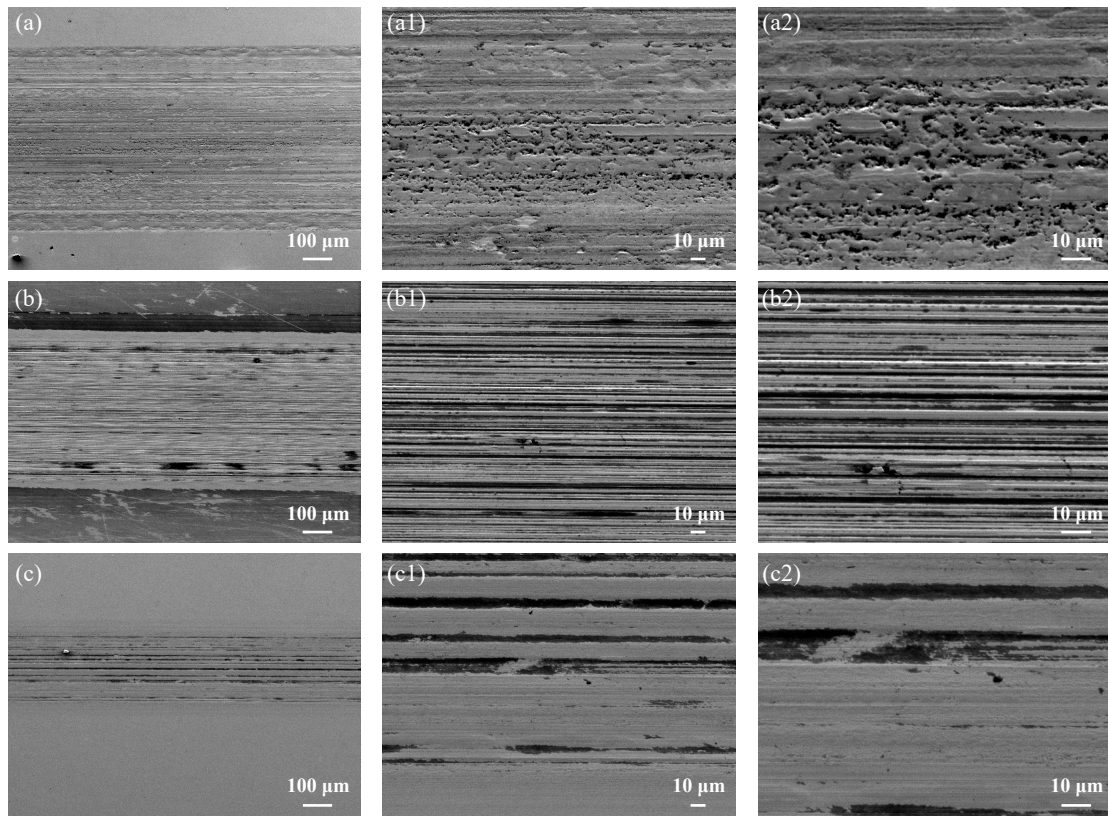


Fig. 9 SEM micrographs of worn surfaces lubricated by different lubricants: (a, a1, a2) WDG; (b, b1, b2) 0.5% DC₁₄-DIPA; (c, c1, c2) 0.5% DC₃₆-DIPA

图 9 不同润滑剂润滑后表面的SEM照片: (a, a1, a2) WDG; (b, b1, b2) 0.5% DC₁₄-DIPA; (c, c1, c2) 0.5% DC₃₆-DIPA

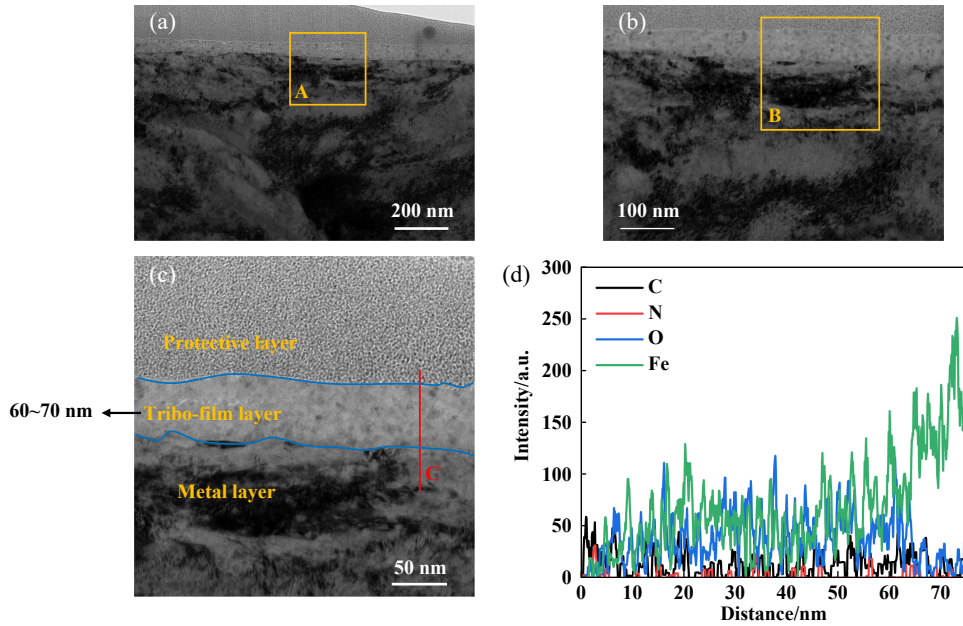


Fig. 10 TEM micrographs of FIB-cut cross-section of the friction film generated by 0.5% DC₃₆-DIPA: (a) TEM micrographs of the abrasion mark; (b) zoomed view of area A; (c) zoomed view of area B; (d) intensity change of EDS element along line C in Figure c

图 10 0.5% DC₃₆-DIPA 润滑表面形成的摩擦膜的 FIB 切割横截面的 TEM 照片: (a) 磨痕的 TEM 照片; (b) 区域 A 的放大视图; (c) 区域 B 的放大视图; (d) 沿 C 图中直线 C 得出的 EDS 元素强度变化

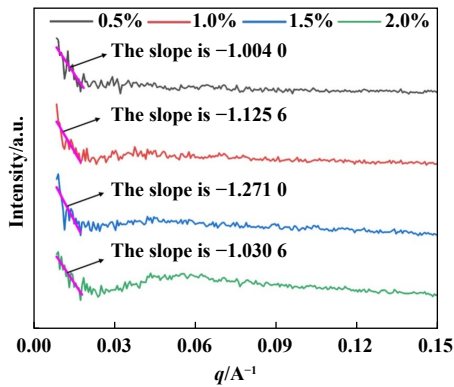


Fig. 11 Small-angle X-ray scattering profiles of DC₃₆-DIPA under different concentrations

图 11 不同浓度的 DC₃₆-DIPA 的 SAXS 散射曲线

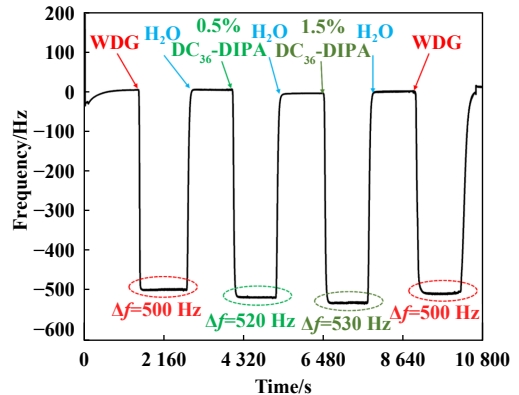


Fig. 12 Changes in frequency of QCM chip gold under different concentrations

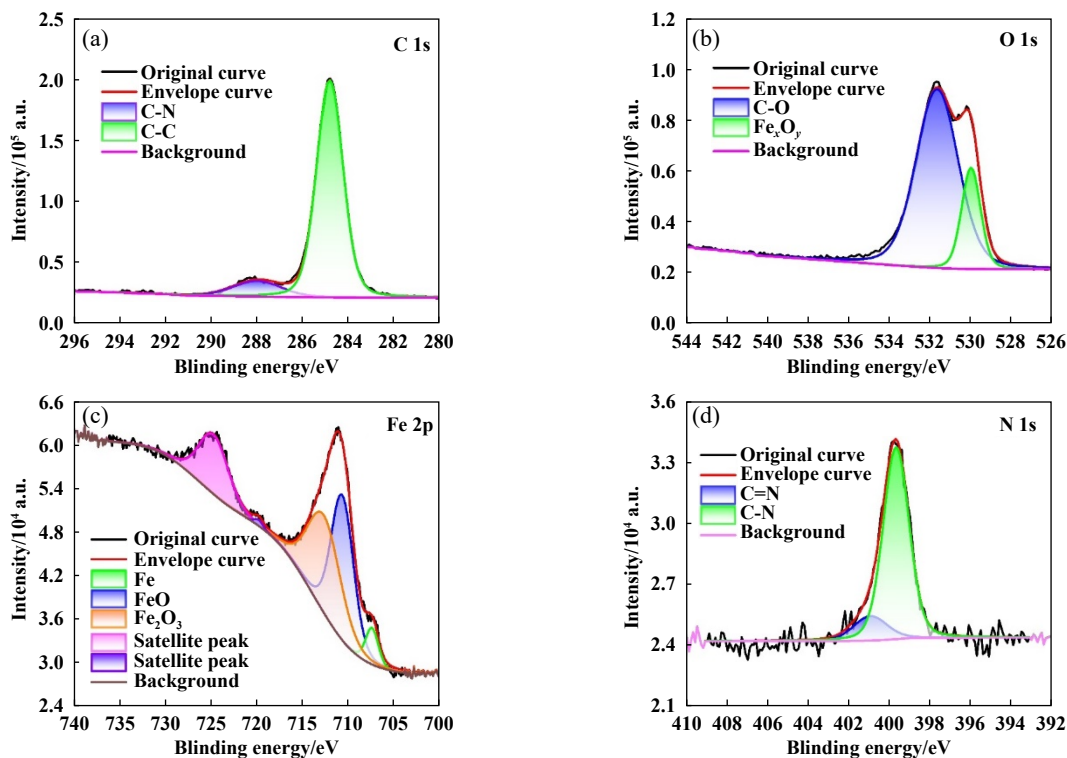
图 12 不同浓度 DC₃₆-DIPA 的 QCM 吸附频率变化

一方面, 试验所用材料为镀金芯片, 金为惰性金属, 难以产生化学吸附, 但本研究中的摩擦学试验采用的是钢材, 其中的铁原子可以与添加剂中极性基团通过化学键产生化学吸附^[36]. 据此推测, 1.5% DC₃₆-DIPA 在产生更强的物理吸附下表现出更高的磨损, 这表明在摩擦的过程中, 化学吸附可能与物理吸附同时存在, 且化学吸附可能更有利于减少摩擦磨损.

2.7 XPS 分析

为了进一步探究含离子液体的水基润滑剂在摩擦副表面形成的摩擦膜的成分, 我们对 0.5% DC₃₆-DIPA 润滑后的磨损痕迹进行了 XPS 能谱的分析. 图 13(a~d) 所示分别给出了 0.5% DC₃₆-DIPA 磨损痕迹处 C 1s、O 1s、

Fe 2p 和 N 1s 的电子结合能. 由图 13(a) 可知, 284.8 eV 处为污染碳及 COO⁻ 的峰, 288 eV 处代表 C-N, 说明离子液体在试块磨痕表面存在吸附; 由图 13(b) 可知, 531.6 eV 处为 C-O 峰, 对应 C 1s 中 284.8 eV 处的峰, 529.9 eV 处为 Fe_xO_y 峰, 说明磨痕表面存在碳氧化物和 FeO/Fe₂O₃ 组成的含氧物质; 由图 13(d) 可知, 399.6 eV 及 401 eV 处为有机 C-N/C=N 峰, 与 C 1s 中 287.6 eV 处峰对应, 说明磨痕表面存在有机氮化化合物的吸附; 由图 13(c) 可知, 707.5 eV 处为 Fe 单质的峰, 710.8 eV 处为 FeO 的峰, 713.1 eV 处为 Fe₂O₃ 的峰, 其余为卫星峰^[37]. 由 XPS 分析结果可知, 试块磨痕表面存在离子液体中

Fig. 13 XPS spectra of the worn surface of 0.5% DC₃₆-DIPA图 13 0.5% DC₃₆-DIPA 润滑表面的 XPS 能谱图

的元素,说明离子液体在润滑过程中参与到了金属表面的物理化学吸附及摩擦化学反应,从而形成了1种由C、O、Fe和N等元素组成的有机边界润滑膜,这与OM、SEM、QCM和FIB-TEM结果一致。

2.8 润滑机制

根据上述OM、SEM、FIB-TEM、SAXS、QCM和XPS的分析结果,提出了二聚酸离子液体(DC₃₆-DIPA)在钢-钢摩擦副表面的润滑机理,如图14所示。在滑动过程中,金属材料内部的低能电子获得能量并逃逸,导致表面充满正电荷,因此,羧酸阴离子很容易吸附在该表面上,优先在滑动表面形成1层自组装的吸附层^[38-40]。在0.5%质量分数下,吸附层由DC₃₆-DIPA囊泡、水和二乙二醇共同组成,中间的水层则不含添加剂分子;在质量分数为1.0%和1.5%下,DC₃₆-DIPA部分转化为胶束,胶束部分代替囊泡进入到吸附层中,水层则由囊泡、胶束、水和二乙二醇分子组成;在质量分数为2.0%下,囊泡完全转化为胶束,吸附层和水层均由胶束、水和二乙二醇分子组成。囊泡和胶束是DC₃₆-DIPA分子在基础液WDG中存在的2种形态,均能够增强基础液的减摩抗磨性能,但囊泡似乎更有利于抗磨性能的增强,而胶束的存在会削弱这种增强作用,表现出在高质量分数($\geq 1.0\%$)下的磨损体积的增

加。此外,水分子的存在是表面物理吸附形成的主要原因,吸附导致了润滑界面上球和盘发生轻微黏附,从而产生少量的黏着磨损^[41]。

在摩擦界面上形成的吸附层对减摩抗磨性能起关键作用,2层吸附层和中间的水层能够利用柔性烷基链的剪切阻力降低沿运动方向的剪切应力,从而有利于降低摩擦系数^[42-43]。与此同时,在摩擦作用下,DC₃₆-DIPA与金属表面发生复杂的摩擦化学反应,从而形成了以铁氧化物为主的边界润滑膜,减少了球和盘的直接接触,降低了磨损。这层润滑膜受到离子液体中极性基团的吸附及润滑剂与摩擦副之间反应的影响。总之,DC₃₆-DIPA在摩擦界面上生成的物理化学吸附膜和摩擦化学反应膜对水基润滑剂的减摩和抗磨性能起主导作用,是其优异摩擦学性能的主要原因。

3 结论

a. 利用不同分子结构的二元脂肪酸和二异丙醇胺合成了4种不同的羧酸铵离子液体,并对其结构进行了表征。初步试验表明,所有的离子液体在水-二乙二醇溶液中均有良好的溶解性和分散稳定性,具备作为水基润滑剂的基本条件。

b. 在室温下,所制备的离子液体能够明显增强水

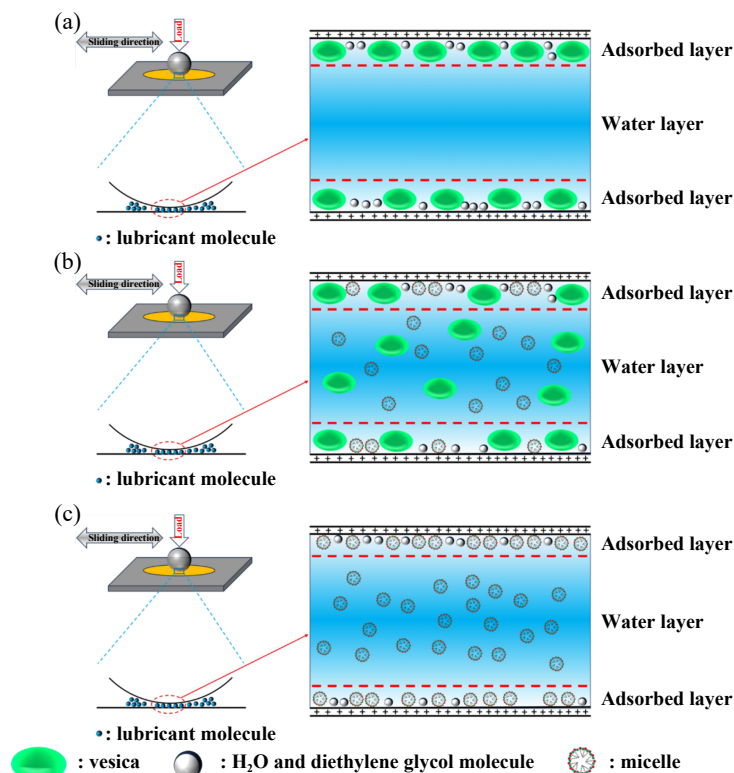


Fig. 14 Lubrication mechanism model diagram of C_{36} -DIPA on steel/steel friction pair: (a) molecular adsorption at vesicle stage; (b) molecular adsorption at vesicle and micelle stage; (c) molecular adsorption at micelle stage

图 14 二聚酸离子液体在钢-钢摩擦副上的润滑机理模型图: (a)囊泡阶段的分子吸附; (b)囊泡和胶束共存阶段的分子吸附; (c)胶束阶段的分子吸附

基润滑体系的减摩性能,但只有 DC_{36} -DIPA增强了基础液的抗磨性能,因此其摩擦学性能最佳; DC_{36} -DIPA在高质量分数($\geq 1.0\%$)下易造成高磨损,低质量分数(0.5%)下则磨损较低; DC_{36} -DIPA具有良好的承载能力,能够显著增强水-二乙二醇的极压性能。

c. SAXS结果表明,浓度可能会影响 DC_{36} -DIPA在基础液中的存在形态,低质量分数(0.5%)下为囊泡,而增加浓度则会发生由囊泡到胶束的转变,抗磨性能变差.而OM、SEM、FIB-TEM、QCM和XPS结果表明, DC_{36} -DIPA不仅能够在金属表面形成自组装的物理化学吸附膜,而且能够与钢-钢摩擦副发生复杂的化学反应,生成以铁氧化物为主要成分的摩擦化学反应膜,这是其在水基润滑体系中主要的润滑机理。

本研究中制备出的二聚酸离子液体在水-二乙二醇中具有优良的摩擦学性能,有望用于具有抗磨性能要求的水-乙二醇液压液体体系中。

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