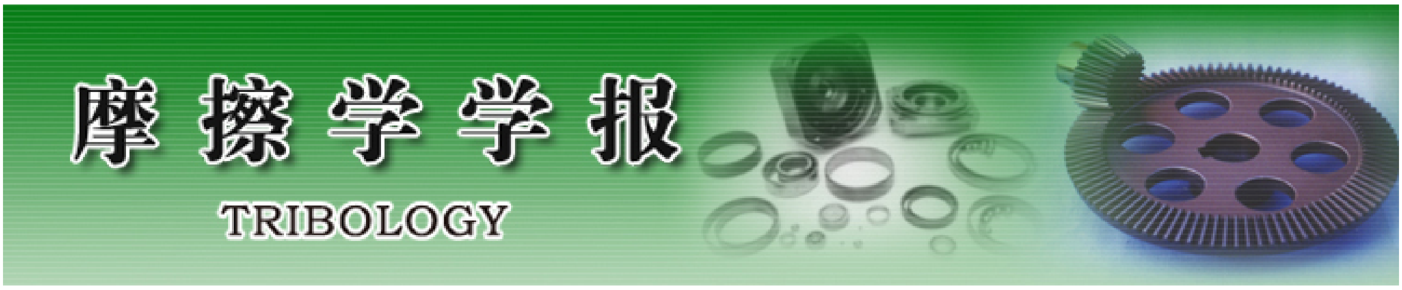

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相对湿度对磁场下45钢摩擦学性能与表面氧化的影响

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摘　要: 电磁制动和电磁弹射是典型磁场条件下的摩擦学系统. 相对湿度对其摩擦学性能和摩擦稳定性有重要影响. 本文中研究了湿度对磁场下45钢自配副的摩擦磨损性能的影响，通过摩擦表面形貌演化分析了摩擦机理和磨损机制的转变，利用能量色散光谱仪(EDS)和X射线光电子能谱仪(XPS)表征了摩擦表面铁元素的氧化程度和氧化形式. 结果表明：随着湿度增加，磁场下45钢自配副的摩擦系数单调增加，磨损率先增加后减少，30% RH为转变点；10%~70% RH范围内随湿度增加，摩擦表面的氧化程度升高，氧铁比变化率下降，表面Fe^{2+}占比增大，OH^-占比减小；70% RH时铁氧化物的水合物析出，氧的存在形式由OH^-向$FeO_x{\cdot}H_2O$转化.

关键词: 磁场; 相对湿度; 摩擦磨损; 氧化膜; 氧化磨损
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Effect of Relative Humidity on Tribological Properties and Surface Oxidation of 45 Steel under Magnetic Field

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Abstract: Under magnetic field conditions, common electromagnetic tribological systems include electromagnetic braking and electromagnetic ejection. Magnetic field promotes the formation of oxidation film on the surface of iron-based friction materials, which changes the friction mechanism and wear mechanism of iron-based materials and improves their wear resistance significantly. Considering practical service conditions of friction couples, the formation of oxide film is closely related to the relative humidity. It is necessary to study the effect of relative humidity on the surface oxidation behavior and friction and wear properties of iron-based friction materials under magnetic field. Being of excellent combination of mechanical and magnetic properties, 45 steel was selected as the research friction material. It was studied that the influence of humidity on the friction and wear properties of 45 steel self-matching pairs under magnetic field. Tribology data were obtained by self-made HY-100 pin-disk friction and wear testing machine. The tests adopt R121 standard of the international Organization of Legal Metrology (OIML) and used the saturated salt solution which had the characteristic of stable vapor pressure to control the ambient humidity of the sealed protection box. Under

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conventional braking mode, the brake pad of maglev vehicle provided braking friction at low speed. Compared with its service conditions, the magnetic field intensity was set to 17.4 kA/m, the contact pressure and the friction speed were kept constant at 1 MPa and 1 m/s respectively. The transformation of friction mechanism and wear mechanism was analyzed through the evolution of friction surface morphology. The degree and state of iron oxidation on the friction surface were characterized by EDS and XPS. The results showed as follow: with the increase of humidity, the friction coefficient of 45 steel self-coupling increased monotonically under magnetic field, however, the wear rate of 45 steel pin first raised to the maximum at 30% RH and then went down until the humidity tended to saturation. When the humidity was at 50% RH, the roughness of surface reached the highest. The surface roughness evolution indicates that the friction mechanism changes from three-body friction to adhesive-fracture mechanism, and the wear mechanism gradually changed from abrasive wear to oxidation wear. With the increase of humidity from 10% to 70% RH, the oxidation content on the friction surface elevated and the oxygen-iron ratio increased from 0.46 to 0.70. However, the effect of increasing humidity on the oxidation rate of 45 steel surface was gradually decreased, and when the air humidity was saturated (>70% RH) was negative. At 70% RH, the iron oxide hydrate was precipitated with the proportion of OH^- decreasing from 77% to 44% and the oxidation state converting from OH^- to $FeO_x{\cdot}H_2O$. The Fe^0 peak gradually decreased until it finally disappears with the increase of humidity, and the surface of the worn pin sample was gradually covered by iron oxide. The proportion of Fe^{2+} on friction surface increased from 49% (10% RH) to 85% (70% RH). The experimental results provided a reference path to control frictional oxidation behavior and improved the frictional properties and environmental stability by the magnetic field.

Key words: magnetic field; relative humidity; friction and wear; oxidation film; oxidation wear

随着超导和电磁技术的快速发展，越来越多的机械设备如电磁制动器、电磁弹射器和电动机等的摩擦副在磁场中运行[1-5]，为了降低摩擦材料的消耗，延长摩擦副的使用寿命，保证摩擦系统在服役过程的稳定性，需要对材料在磁场下的干摩擦性能进行系统研究.

施加磁场可显著改善钢的摩擦学性能. 在磁场条件下铁基材料摩擦副的磨损减少，摩擦系数及其波动性减小. 董祥林等[6]和简小刚等[7]研究了磁场对中碳钢的滑动摩擦和磨损的影响，发现磁场使中碳钢的氧化磨损比例增加，从而导致磨损减少. Chin等[8-9]和Zaidi等[10]根据多组试验数据，初步建立了磁场对铁磁性材料摩擦学性能影响的数学模型. 磁场对摩擦磨损的影响机制主要包括3个方面：首先磁场能促进摩擦表面形成稳定的氧化膜，显著改善耐磨性能[11]；其次在磁场作用下，磨屑吸附于摩擦表面并参与摩擦过程，形成三体摩擦机制[12]；最后磁场也能影响材料次表面的组织结构，促进位错向摩擦副接触面迁移[13-14].

摩擦学性能取决于材料的力学性能和其服役工况. 服役工况包括接触压力，摩擦速度，还包括摩擦副的服役环境，如温度、湿度和真空等因素. 相对湿度与铁基材料的摩擦学特性密切相关. 盛选禹等[15]认为对于金属摩擦副，水在接触界面处形成的弯月面增加了附着力，增大了摩擦系数；Baets等[16-17]认为在高湿度时水分在表面之间凝结形成润滑膜，并且在表面发生了更为复杂的氧化反应，生成氢氧化铁和氧化铁水合物，磨损表面的氧化速率降低，磨损率下降；Goto等[18]研究了湿度对制动盘材料摩擦学性能的影响，结果表明当湿度增加时，制动器的磨损显著减少，其可能原因是摩擦界面形成的水膜起到了润滑的作用. 另外，研究表明高湿度会加速铁、铝和铜等金属的磨损，Goto和Buckley等[19]研究了湿度对铁、铜等7种不同金属摩擦系数和磨损的影响，发现即使少量的水蒸气也会干扰氧气在摩擦表面上的吸附，增加摩擦表面间的黏着接触. Endo等[20]的试验也表明湿度增加会加速金属铝裂纹的萌生与扩展，进而增加了其磨损.

综上，磁场之所以能显著改善导磁材料的摩擦学性能，主要是因为磁场能够促进摩擦表面氧化反应，在摩擦面形成稳定的铁氧化膜，改变了铁基材料的摩擦机理和磨损机制. 铁的氧化行为和相对湿度密切相关[21]，但相对湿度如何影响磁场下铁基材料的摩擦氧化行为，进而如何影响其摩擦磨损性能的相关研究尚未见报道. 45钢具有优异机械性能和导磁性能，有良好的研究基础[22-24]. 本研究选用45钢自配副为研究对象，分析不同湿度对磁场下45钢摩擦系数和磨损率的影响，采用摩擦表面多尺度分析方法，揭示了不同湿度条件下45钢摩擦机理和磨损机制的转变，最后利用能量色散光谱仪(EDS)和X射线光电子能谱仪(XPS)表征总结了不同湿度下摩擦表面铁元素的氧化程度和氧化形式. 试验结果将为磁场调控摩擦氧化行为，进而改善摩擦性能和环境稳定性提供可供参考的路径选择.

1 试验部分

试验选择正火态45钢为摩擦材料，45钢具有优异机械性能(显微硬度为HRC 28，抗拉强度为600 MPa，屈服强度为355 MPa)[25]和导磁性能(铁磁性材料，最大磁导率为583)，是研究磁场干涉摩擦行为的典型材料. 45钢的主要成分为Fe，其他少量元素的质量分数为C 0.42%～0.50%，Cr ≤0.25%，Mn 0.50%～0.80%，Ni ≤ 0.25%，P ≤0.035%，S ≤0.035%，Si 0.17%～0.37%. 摩擦接触方式为销/盘接触，摩擦接触面积为78.5 mm^2，摩擦盘的回转半径为72.5 mm.

试验采用自制的HY-100型销盘式摩擦磨损试验机，如图1(a)所示. 试验机装有外部铜质线圈，线圈通直流电后产生恒磁场，磁场强度和电流正相关，磁场方向垂直于销盘摩擦副接触表面. 为了控制摩擦过程中的相对湿度，在试验机上加装了密封保护箱和洗气瓶，采用国际法制计量组织(OIML)的R121标准，利用饱和盐溶液具有稳定蒸汽压的特性，通入气体使其达到目标湿度，进而控制密封保护箱的环境湿度，通过SMART-AR847型号的湿度传感器，监测密封箱中的湿度变化. 由于在摩擦过程中出现温升会引起湿度的波动，因此摩擦试验环境的实际相对湿度控制在±5°范围区间.

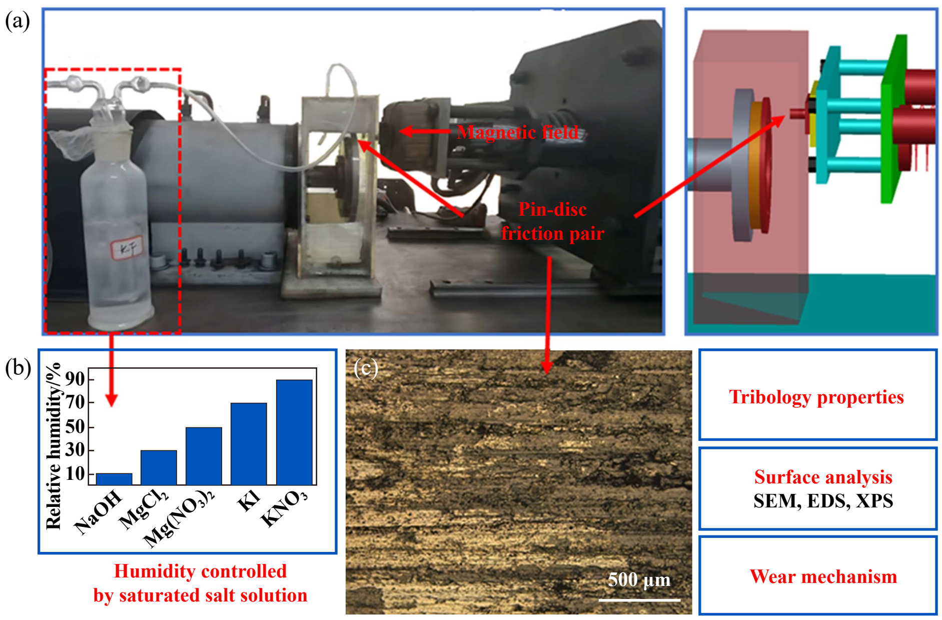

Fig. 1 (a) HY-100 pin-disc magnetic field friction and wear tester; (b) humidity controlled by salt solution; (c) metallographic diagram of friction surface.

图 1 (a) HY-100 销盘式摩擦磨损试验机，(b) 不同湿度下盐溶液的选取，(c) 摩擦表面金相图

试验前对销和盘试样进行预处理，首先用多种砂纸依次打磨摩擦表面，其次用酒精和丙酮擦拭，去除表面的氧化膜与油膜，使试验前各组摩擦副的初始表面状态与粗糙度保持一致，最后对试样进行退磁处理. 摩擦试验选取10%、30%、50%、70%和90%的相对湿度，对照饱和盐溶液的平衡相对湿度表，分别采用NaOH、$MgCl_2$、$Mg(NO_3)_2$、KI和KNO_3的饱和溶液实现相应湿度[26-27]；磁场强度为17.4 kA/m，根据45钢的磁化曲线[28]，该磁场强度下45钢为完全磁化状态；在常规制动模式下磁浮车用闸片在低速时提供制动摩擦力，对照其服役工况，接触压强设为1 MPa，摩擦速度为1 m/s. 为保证试验的重复性和可靠性，重复5次试验取平均值为试验结果. 试验材料的摩擦系数μ和线性磨损率ω的计算方法如公式(1)和公式(2)所示：

$$\mu(M_1-M_0)/F_nR \tag{1}$$

$$\omega=\Delta G/vt \tag{2}$$

式中：M_1为摩擦扭矩；M_0为空载扭矩；R为回转半径；F_n为实际载荷；$\triangle G$为试验前后的质量差；v为摩擦速度；t为摩擦时间.

用光学显微镜(OM, 型号: Leica-DMI 8C)观察销试样摩擦表面形貌；白光干涉轮廓仪(WLIP; 型号: Nanofoucs-μSurf explorer)记录摩擦表面轮廓，测量磨痕深度和宽度，计算得到摩擦后的表面粗糙度；扫描电子显微镜(SEM; 型号: Tescan-Vega3 SBH)观察摩擦后表面的微观形貌，能量色散光谱仪(EDS)表征摩

擦后表面铁Fe元素和氧O元素表面分布和相对含量；X射线光电子能谱仪(XPS; 型号: K-alpha-Thermo Scientific)进一步分析摩擦后表面各元素的化学状态.

2　结果与分析

2.1　摩擦学性能

在磁场下45钢互配副的摩擦系数随时间动态变化，且不同湿度下的摩擦系数曲线具有较高的相似性，如图2(a)所示. 在低湿度下45钢的摩擦系数在前期的磨合后逐渐平稳，而在高湿度下，经历初期的磨合过程后摩擦系数仍未完全稳定，并呈现出逐渐上升的趋势. 计算不同湿度下摩擦系数平均值如图2(b)所示. 45钢的摩擦系数平均值和相对湿度密切相关：随着相对湿度的增加，摩擦系数呈正相关单调变化；且30%~50%和50%~70%湿度下摩擦系数变化值要远大于10%~30%和70%~90%. 综上，摩擦系数时序曲线和平均值在不同湿度条件下的区别显著，这表明不同湿度下45钢的摩擦学行为也会有所不同.

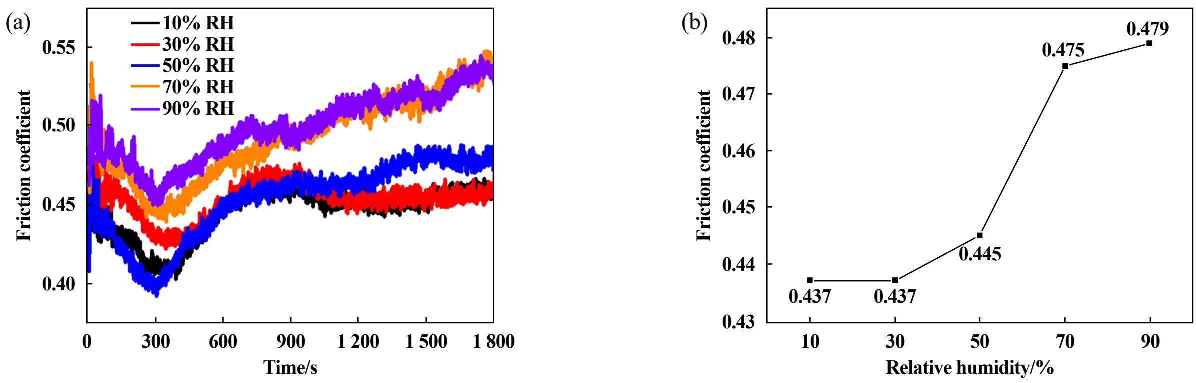

Fig. 2　(a) Variation of friction coefficient at different humidity; (b) average friction coefficient in stable stage

图 2　(a)不同湿度下摩擦系数动态曲线图; (b)稳定摩擦阶段摩擦系数均值

根据5组重复试验数据，不同湿度下45钢自配副的磨损率平均数和误差如图3所示. 45钢磨损率随湿度增加的变化趋势和摩擦系数不同，表现为先增加后减少，当湿度为30%时，磨损率达到最大. 这表明30% RH时45钢磨损机制发生了转变，因此需要进一步分析不同湿度下45钢摩擦后的表面形貌和氧化状态.

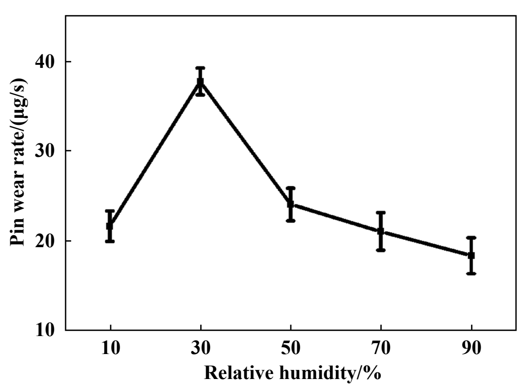

Fig. 3　Effect of humidity on wear rate of pin samples under magnetic field

图 3　磁场下湿度对销试样磨损率的影响

2.2　表面分析

摩擦材料的表面状态和摩擦磨损性能密切相关，为了分析相对湿度对摩擦机理和磨损形式的干涉机制，需要利用光学显微镜、白光干涉轮廓仪和电子显微镜等多种方法多尺度观察45钢摩擦后的表面形貌.

磨损过后销试样宏观形貌的显微镜照片(上)和光学显微镜照片(下)，如图4所示.从销试样表面形貌的显微镜照片可以看出，随着湿度的增加，销表面的黑色区域从前端向后端，从中间向两边进行扩张，黑色区域应为铁的氧化混合物[25]. 摩擦表面大致可以分为3个区域，区域1的磨损最严重，区域2次之，区域3的磨损最轻. 结合销试样光学显微镜照片分析，低湿度下摩擦表面犁沟多且浅，黑色区域占比小；随着湿度的增加，黑色氧化区逐渐扩展，犁沟数量减少，但深度增加；高湿度下摩擦表面几乎完全被黑色氧化混合物所覆盖. 随着湿度的变化，45钢表面氧化状态呈现出肉眼可见的变化，且氧化区域的发展方向表现出从前端向后端，从中间向两边的趋势.

不同湿度下摩擦后的销试样表面三维形貌图(上)和剖面图(下)，如图5所示. 低湿度时磨损表面的犁沟窄而浅，最大深度为11.8 μm，数量虽然较多，但峰谷落差小，因此粗糙度较低，10%湿度时为2.40 μm；随湿度增加，摩擦表面犁沟变宽变深，数量变少，

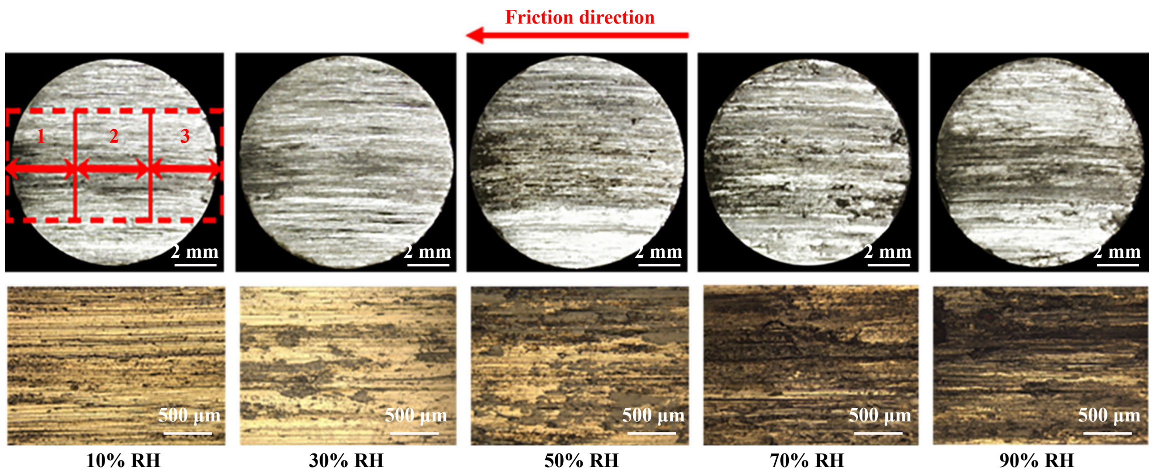

Fig. 4 SEM micrographs (top) and OM images (bottom) of worn pin surfaces

图 4 磨损过后销试样宏观形貌的显微镜照片(上)和光学显微镜照片(下)

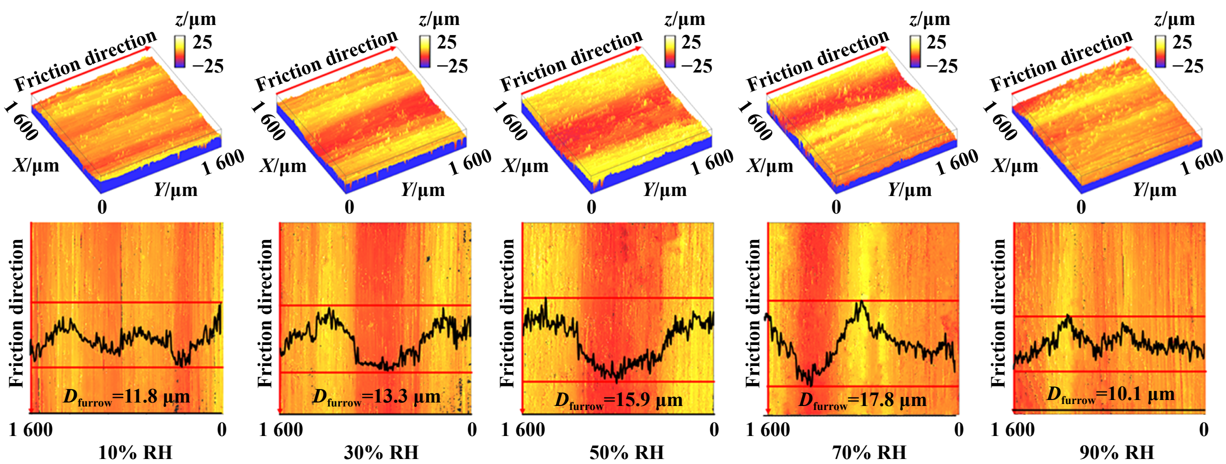

Fig. 5 Three-dimensional morphologies (top) and profiles (bottom) of worn pin surfaces

图 5 磨损过后销试样表面三维形貌图(上)和剖面轮廓图(下)

50%湿度时粗糙度达到最大，为6.44 μm；当湿度继续增加时，随着铁氧化物面积的增加，犁沟先变窄后变浅，表面逐渐平整，90%湿度时粗糙度下降至2.15 μm. 图6所示为5组统计数据计算的销试样平均粗糙度点线图，随着相对湿度增加，销试样表面粗糙度在50%时发生突变.

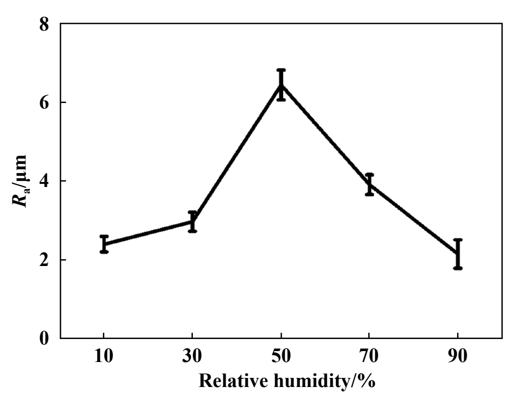

Fig. 6 The average roughness of worn pin surface

图 6 磨损过后销试样表面粗糙度平均值

为了观察不同湿度下磨损后45钢销试样亚表层组织形貌和晶相的变化，沿平行于摩擦方向制备了销试样45°斜剖样，在光学显微镜下亚表面的金相结构如图7所示. 由图7可知，45钢亚表层在摩擦力和磁吸力的作用下发生了塑性形变：低湿度时塑性变形层宽度较大，中高湿度下塑性变形宽度较小. 变形层的宽度变化和磨损形式的变化相关：低湿度下未形成氧化层，摩擦力直接作用于45钢亚表层的晶粒，导致力传导深度增加，形变层变宽；高湿度下，摩擦力作用于氧化层，导致摩擦力在次表层中传导深度下降，形变层宽度变窄.

为了研究销试样表面微观形貌在不同湿度下的演化规律以及相对湿度对表面氧化程度的影响，我们利用SEM表征了不同湿度下摩擦后销试样表面的微

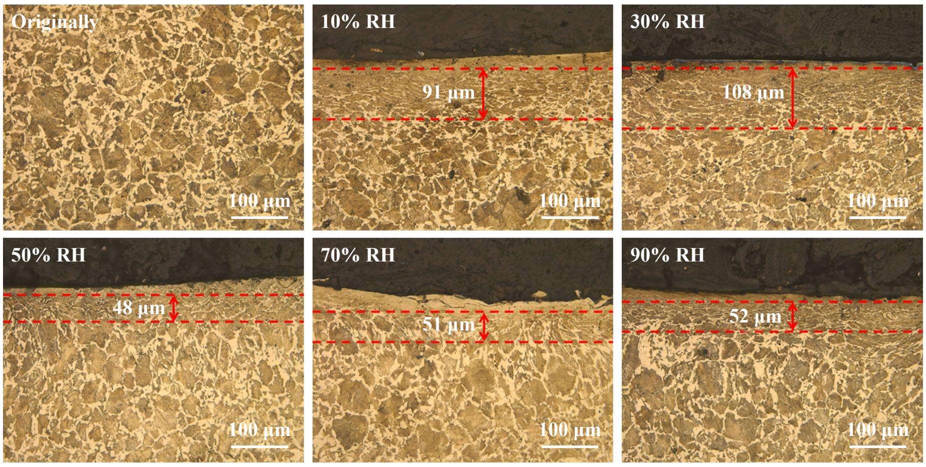

Fig. 7　The microstructure metallography of pin samples
图 7　销试样磨损表面次表层组织金相图

观形貌，并对其进行了EDS元素分析，如图8所示. 通过销试样表面微观形貌的SEM照片观察发现：10% RH时摩擦表面划痕浅且多，痕迹清晰，基本观察不到附着物，推测其磨损形式应为磨粒磨损；30% RH时表面划痕变粗，且能观察到有大颗粒的磨屑，此时磨屑开始发生氧化并团聚，而表面仍未氧化，氧化磨屑切削未氧化表面，因此在30% RH时磨损率最大. 50% RH时摩擦表面开始出现零星和小片的附着物[29]，但仍可以观察到划痕，由于附着物分散分布且仍存在，造成50% RH时粗糙度最高；此时的表面部分氧化并发生黏着，磨损形式变为磨粒磨损和氧化磨损，摩擦学行为也从三体模型过渡到黏着-断裂模型，此时摩擦系数升高. 高湿度下磨损表面几乎被片状氧化物覆盖，表面更加平整，真实摩擦面积增加，因此摩擦系数会随着摩擦时间的增加而增加；同时能观察到因片状氧化物脱落而出现剥落坑，而这会引起摩擦系数的波动，从而解释了图2(a)中高湿度下摩擦后期摩擦系数曲线发生较大振动的原因，此时磨损形式为氧化磨损.

EDS元素分析结果表明，摩擦表面的主要化学组成为铁和铁氧化物，氧元素分布(红点)和铁元素分布

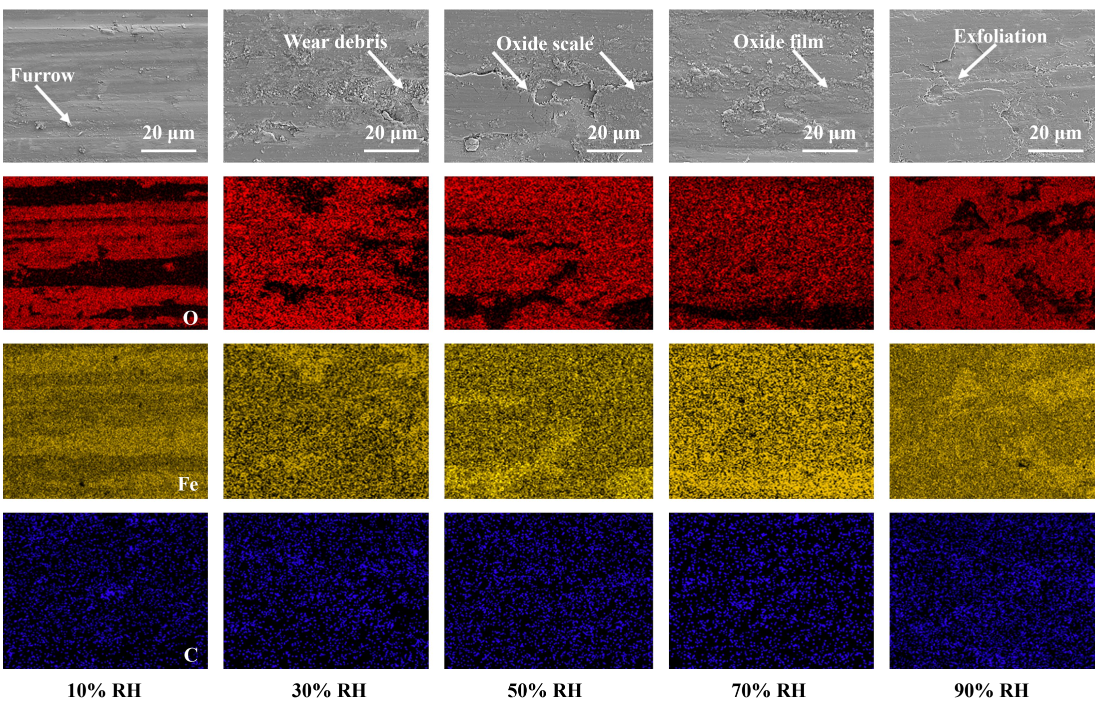

Fig. 8　SEM micrographs and EDS mapping analysis of worn surfaces.
图 8　磨损表面形貌的SEM照片及EDS图谱

(黄点)的面扫描结果如图8所示. 随着湿度增加，EDS面扫描中氧分布的连续性逐渐增加，在70% RH时达到最大，这表明随湿度增加，摩擦表面氧化逐渐充分. 10%~70% RH时EDS氧分布图存在一些无氧分布区域，对照相应的SEM照片可以观察到其表面平整仅颜色有所变化，应为表面尚未氧化所致；对比观察90% RH时出现边缘清晰的有氧/无氧边缘，铁分布图中此处铁元素含量高，结合SEM照片观察到相同位置出现层状剥落结构，该处划痕应是由于氧化物剥落而产生.

相对于O元素和Fe元素的变化，不同湿度下O和Fe的原子比能更好地反应表面氧化程度. 由表1可以看出，氧和铁的原子比值变化趋势和EDS面扫描氧分布的变化趋势相同，但90% RH时下降至0.81，这可能是由于氧化层剥落所致. 值得注意的是，O/Fe原子比的增长率是逐步降低的，由15.15%下降到4.94%，然后负增长为−4.71%，这表明随着湿度增加，其对于45钢表面氧化反应速率的作用是逐渐降低的，当空气湿度饱和时(>70%)为负作用.

90% RH条件下销试样摩擦后表面的XPS总谱如图9(a)所示，结合能反映了原子间的紧密程度，从小到大依次可以观察到Fe、O和C的特征峰，分别为722.5 eV、709.6 eV处的Fe 2p1与Fe 2p3，531.1 eV处的O 1s以及284.8 eV处的C 1s. 为了深入分析不同湿度下销表面各元素的实际化学环境，对O、Fe和C峰放大展示，如图9(b~d)所示. 从O 1s光谱的主峰位置来看，531.3±0.2 eV是羟基氧的典型特征峰；随着湿度的增加，O峰逐渐变宽，在高湿度(70%~90%)下，在主峰左侧O 1s峰(532.8 eV)和主峰右侧O 1s峰(528.6 eV)面积都显著增加，这表明在高湿度下铁的氧化形式发生了变化，出现了新的氧化产物. 对照XPS标准图谱，此处氧的化学形式分别应为羟基和氧双键，这可能是由于高湿度条件下45钢表面形成水膜，氧气无法和铁元素直接接触，因为水供给充足而氧供给有限，导致Fe氧化过程中的中间产物FeOOH无法进行脱水反应形成铁氧化物. Fe 2p可以分为2个峰值，分别对应于Fe 2p1轨道和Fe 2p3轨道[30]，同时观察到在高湿度条件下，Fe 2p

表 1　摩擦表面能谱分析元素统计表

Table 1　Atomic fraction of elements on worn surfaces

Relative humidity/%	Atomic fraction/% C	O	Fe	O/Fe	The growth rate of O/Fe
10	20.48	31.59	47.93	0.66	−
30	22.54	33.35	44.11	0.76	15.15
50	17.04	37.22	45.74	0.81	6.58
70	17.48	37.99	44.53	0.85	4.94
90	17.23	34.09	48.97	0.81	−4.71

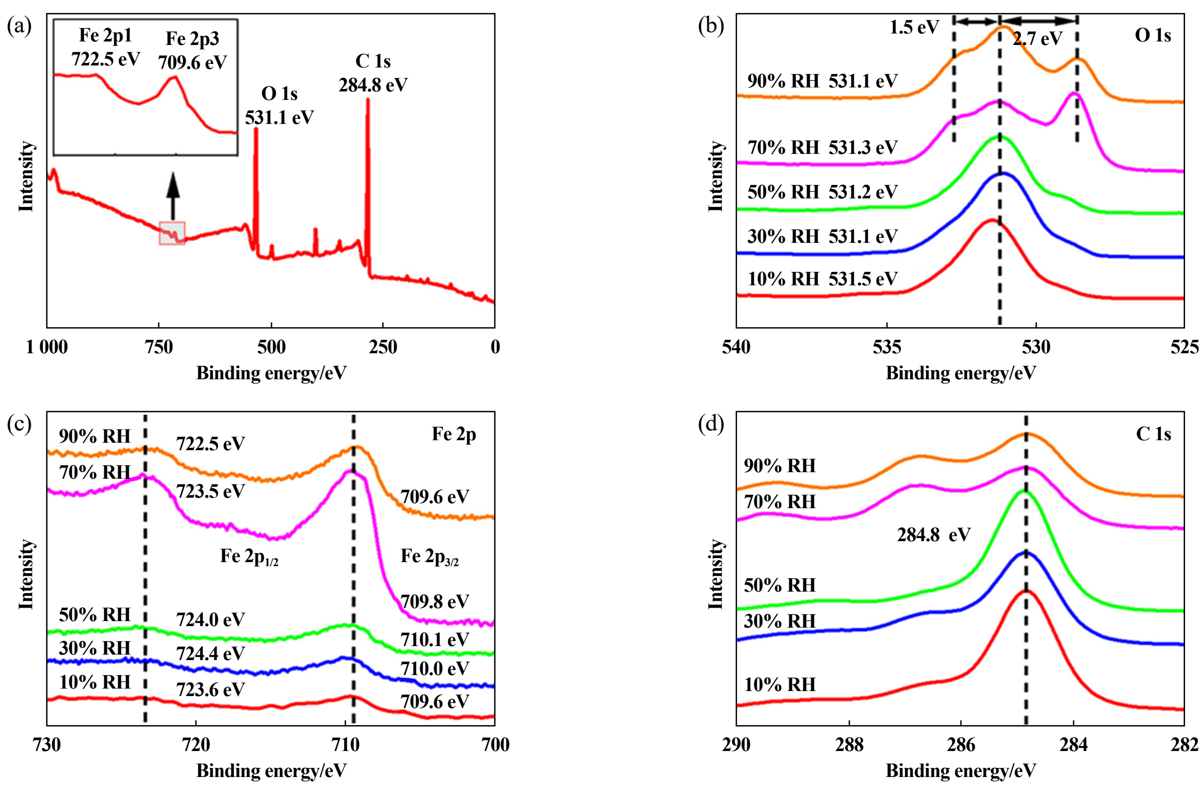

Fig. 9　XPS spectra for worn pin surfaces: (a) full spectrum; (b) O 1s; (c) Fe 2p; (d) C 1s

图 9　摩擦后销试样表面的XPS谱图：(a) 总谱；(b) O 1s；(c) Fe 2p；(d) C 1s

峰形发生了较大的变化，表明此时的Fe元素化学环境有了较为显著的变化. 从C 1s光谱来看，286.6±0.2 eV的新峰表明45钢中少量碳在高湿度下也参与了反应.

XPS结果表明销试样摩擦表面Fe的氧化程度和氧化形式都随着湿度增加而变化，为了进一步分析其演化规律，量化区分O和Fe原子的处于不同化学状态的相对比例，本文中参考不同化学环境下O和Fe的标准峰数据，分别对O和Fe的特征轨道峰进行了分峰拟合处理，如图10所示. 对照XPS图谱O 1s标准峰位置，羟基氧主峰(OH^-)左侧应该为结合水中的O 1s峰(H_2O)，主峰右侧推测为铁氧化物的O 1s峰(Fe_3O_4)，且两峰面积同步增加，这表明70% RH时，大量铁氧化物的水合物析出，氧的存在形式由OH^-向$Fe_3O_4 \cdot H_2O$转化[31-33]，OH^-所占比例由77%降低至44%. 随着铁氧化物含量的提升，Fe 2p3峰也发生较大变化. Fe 2p3轨道峰被分为Fe^0，Fe^{2+}和Fe^{3+}，分峰位置分别位于706.0、709.6 和712.8 eV[34-36]. 由图10可知，Fe^0峰随着湿度增加而逐渐减少直至消失，这表明随着湿度的增加，磨损后的销试样表面逐渐被铁氧化物覆盖. 并且Fe 2p3峰发生红移现象，且随着湿度增加铁的平均氧化价态随之降低. 根据分峰面积计算，Fe^{2+}所占铁元素化学形态比例由49% (10% RH)升高至85% (70% RH).

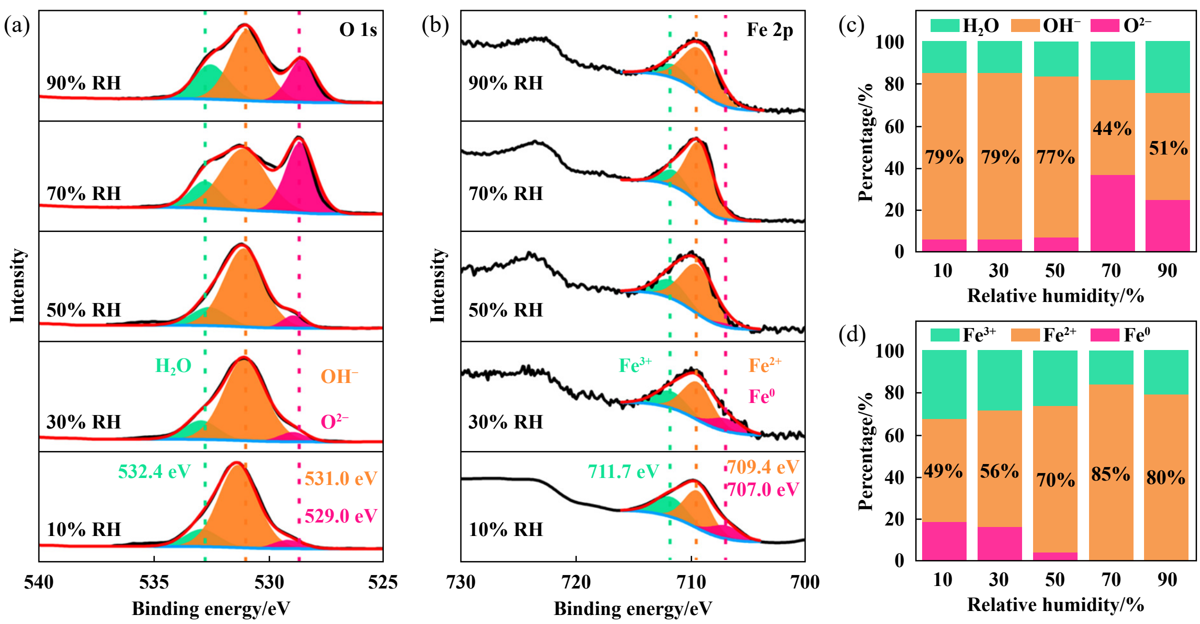

Fig. 10　XPS peak-fitting analysis of (a) O 1s and (b) Fe 2p on worn surfaces; content change chart: (c) O 1s and (d) Fe 2p

图 10　磨损试样表面XPS分峰拟合分析 (a) O 1s和(b) Fe 2p; 化学形态占比图: (c) O 1s; (d) Fe 2p

3　结论

a. 随着相对湿度的增加，磁场下45钢互配副的摩擦系数呈正相关单调变化，磨损率先增加后减少，30% RH为转变点.

b. 三维形貌统计结果中50% RH时摩擦表面粗糙度最高，摩擦表面同时观察到存在氧化物和划痕，这表明50% RH时磁场下45钢的摩擦机理和磨损机制发生变化；综合考虑多尺度下的摩擦表面形貌，推测摩擦机理从三体摩擦机理过渡到黏着-断裂机理，磨损机制由磨粒磨损逐步转化为氧化磨损.

c. 相对湿度影响摩擦表面的氧化程度，EDS结果表明，摩擦表面的氧铁比随湿度增加而升高，但其变化率逐渐降低，当空气湿度饱和时(>70%)为负作用.

d. 不同湿度下的氧化产物也不同，XPS结果表明，摩擦表面Fe^{2+}所占比例由49% (10% RH)升高至85% (70% RH)，且在70% RH时铁氧化物的水合物析出，氧的存在形式由OH^-向$FeO_x \cdot H_2O$转化.

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