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10 m 长管道内壁类金刚石薄膜沉积及性能*

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摘要: 工业生产过程中管道内壁经常受到输送物质的腐蚀和磨损,对管道内壁进行涂层防护十分必要,而目前鲜有大长径比管道内壁镀膜的报道,且缺乏对大长径比管道内壁膜层性能的研究。采用等离子体增强化学气相沉积(PECVD)技术在直径100 mm、长10 m管道内壁沉积类金刚石(DLC)薄膜,并研究管道内工作气体的等离子体放电辉光光谱、膜层表面亮度、水静态接触角、硬度、摩擦因数和拉曼光谱等。结果表明:管道内等离子体光谱显示管内等离子体中有 Ar^+ 和乙炔分解成的 C_2 、H和CH;膜层表面的亮度 L^* 最高达到37.4和色差 ΔE^* 最大1.9,膜层拉曼光谱结果表明靠近管道两端和中间位置膜层的 I_D/I_G 均匀,膜层水静态接触角显示靠近管道两端的膜层接触角略小;靠近管道两端膜层硬度相比中间位置的膜层硬度高,并且磨损测试中膜层均未出现破损剥落,膜层具有高的耐磨性。试验实现了在大长径比的管道内壁沉积耐磨损的DLC膜层,为长管道内壁均匀镀膜提供了理论支持和技术指导。

关键词: 等离子体增强化学气相沉积(PECVD);长管道;内壁镀膜;类金刚石

中图分类号: TG156; TB114

Deposition and Properties of DLC Film on the Inner Wall of a 10-m-Long Stainless-steel Tube

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Abstract: During industrial production, the inner wall of a tube is often corroded and worn by the conveying material; therefore, it must be protected. Currently, research on the coating of the inner wall of a tube is limited, and research regarding the performance of the inner wall of a large-aspect-ratio tube is lacking. Thus, this study attempts to solve the problem of a diamond-like carbon(DLC) film on the inner wall of a long tube. Herein, the internal surface of a 304 stainless-steel tube, which is 10 m long and 100 mm in diameter, is coated with DLC using plasma-enhanced chemical vapor deposition(PECVD). The hollow cathode discharge of gas into the tube, brightness of the film, contact angle, surface hardness, friction factor, and Raman spectroscopy results are characterized using different tests. During the preparation of the inner coating, the plasma spectrum in the 300–800 band at position 3 of the pipe is analyzed using a spectrometer and optical focusing platform. After the film is prepared, its surface brightness is measured using a color difference meter to determine the L^* , a^* , and b^* values of the film, where L^* represents the brightness, a^* represents red and green, and b^* represents yellow and blue. Scanning electron microscopy(SEM) is used to observe the section morphology of the film. The surface roughness of the film is measured using atomic force microscopy(AFM). A friction and wear-testing machine is used to test the wear resistance of the film. A GCr 15 ball with a diameter of 3 mm is selected as the grinding pair, and the experimental parameters of load, rotational speed, and test time are set as 3 N, 200 r/min, and 120 min, respectively. A nanoindentation hardness

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instrument is used to measure the nanohardness of the film. A measuring meter for the static contact angle of water is used for measurement; the measurement requires 2 μl of each drop. Each sample is tested three times, and the average value is calculated as the final test result. The film is measured by a laser Raman spectrometer using a 532 nm laser source from 800 to 2 000 cm^{-1} , and a Gaussian curve is employed to fit the Raman spectrum. The results demonstrate that in the coating process of the inner wall, the first step is the entry of Ar gas into the pipe for glow discharge cleaning. Ionization and excitation occur during the process of Ar gas discharge, thereby producing Ar^+ and Ar^0 ; additionally, C_2 , H, and CH decompose from acetylene. The SEM results of the cross-sections of the films at different positions reveal that the cross-section of the film is uniform and dense, with no cracks, pinholes, or other defects. Further, no cracks are observed in the binding transition between the film and matrix, thickness of the film at different positions, and thickness of the film near the ends of the pipeline, thus indicating that the plasma density in this region and the deposition rate of the film are high. The largest L^* and ΔE^* values of the film are 37.4 and 1.9, respectively. The AFM results demonstrate larger bulges on the film surface at positions 3 and 7, which result in high surface-roughness, and uniform distribution of the bulges at positions 3 and 9. The water contact angles of the films are smaller at both ends. The results of the wear tests indicate that the friction factor of the film decreases in the first 20 min from 0.225 to approximately 0.175. For a prolonged time, the friction factor remains stable at 0.17. The hardness of the film near the ends of the pipe is slightly higher. Further, no damage or film-peeling is noted in the pin-on-disk test, and the film is characterized as having a good wear resistance. The Raman spectra of the films at different positions reveal that in contrast to positions 1 and 9, the G peaks at positions 3 and 7 exhibit a shift toward the peak level, thus indicating a high stress in the film at both ends. Considering the distribution trend of I_D / I_G in the film, a low ratio of I_D / I_G is determined at both ends of the tube. The glow discharge and DLC deposition in the long tube with a large length ratio provides theoretical support and technical guidance for the uniform coating of the walls of long pipes.

Keywords: plasma enhanced chemical vapor deposition (PECVD); long tube; inner coating; diamond-like carbon

0 前言

管道作为工业传输系统的重要组成部分,广泛应用于石油化工、海洋工程、供水、污水处理等领域^[1]。但管道内输送的材料中含有酸、碱等物质,管道内壁发生腐蚀、磨损,带来了重大的经济损失^[2]。因此,研究人员采用了一系列涂层技术来解决这一问题,如喷涂^[3-4]、电镀^[5]、等离子体涂层技术^[6-8]等。在众多涂层技术中,采用等离子体涂层技术在管道内沉积类金刚石(Diamond-like carbon, DLC)薄膜取得了较好的效果,其原因是 DLC 薄膜具有高硬度、高化学惰性、低摩擦因数和磨损率,以及优良的耐腐蚀性^[9-11]。

目前,研究人员主要研究的是在长度为 20~200 cm 的管道内壁镀膜,且管道内壁膜层不均匀。对于大长径比(长度大于 2 m)的管道内壁均匀镀膜更是具有极大挑战。例如, PILLACA 等^[12]研究了在直径 10 cm、长 2 m 管道内,不同气体放电时等离子体的温度和进气与抽气形式对管内膜层厚度分布的影响。结果表明,进气与抽气方式直接影响膜层厚度的分布。WANG 等^[13]报道了在直径 56 mm、长 56 cm 的管道内进行沉积 DLC 薄膜,发现在管道一端进气另一端抽气时,膜层的厚度随进气方向逐渐减小。BABA 等^[14]在长 1 m、直径 35 和 20 mm

的管道内沉积 DLC 膜层,膜层的厚度随进气方向先增大后减小。以上研究显示出膜层在管内不均匀的问题,分析主要原因有:①管道较长,采用现有方式向管道内部导入气体,气体在管道内部分布不均匀,导致管道内气体放电时等离子体均匀性差,在管内制备的膜层厚度和性能差异较大,影响了管内壁膜层的使用效果。②管道过长过大时,由于地电极距离管道内部较远,管道内等离子体在阴极和阳极之间传输效率低,导致管道内部等离子体密度极低,甚至无法启辉,严重影响了管道内部镀膜,制约了长管道内部镀膜技术的发展与应用。

本文采用自主研发的长管道内壁镀膜设备与电源,利用等离子体增强化学气相沉积技术(Plasma enhanced chemical vapor deposition, PECVD)研究长 10 m、直径 100 mm 管道内壁 DLC 涂层沉积,探究影响 10 m 长管道内壁膜层沉积及其性能的因素。研究管道内工作气体辉光放电的光谱以及不同位置膜层的亮度、粗糙度、硬度、耐磨性、接触角和薄膜微观结构,为实际工业生产奠定理论基础。

1 试验准备

1.1 试验装置样品制备

试验装置为自行研发设计的长管道内壁镀膜设备,由机械泵、分子泵、真空腔室、管道组成。被

镀管道作为真空室的一部分。设备可对直径 50~300 mm、长 1~12 m 的管道内壁进行镀膜。镀膜电源为实验室自主研制的高压脉冲镀膜电源, 如图 1 所示, 电压 0.4~10 kV、频率 0~1.2 kHz。采用直径 100 mm、长 10 m 的 304 不锈钢管。硅片试样为 10 mm×10 mm 的样片, 用于检测 DLC 薄膜的厚度。不锈钢试样切割成 20 mm×20 mm 抛光后, 在室温下用无水酒精超声清洗 20 min, 再用去离子水冲洗干净, 吹风机吹干后, 在 10 m 管道内每间隔 1 m 放置一片, 编号 1~10。工作气体采用高纯度 Ar 气、高纯度乙炔、四甲基己硅烷(TMS), 纯度为 99.99%。镀膜工艺开始前真空室初始真空为 1.0 mPa, 之后充入 200 cm³/min 的 Ar 气, 并进行 30 min 的 Ar 离子清洗, 清洗完成后通入 20 min 的 20 cm³/min TMS 沉积粘结界, 最后通入 1 h 的 100 cm³/min 乙炔和 100 cm³/min Ar 气制备 DLC 膜层。

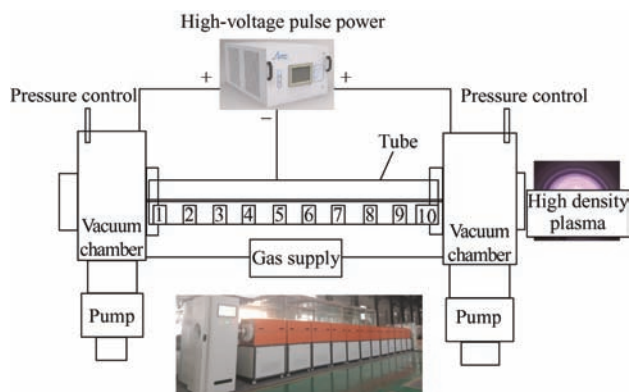


图 1 长管道内壁镀膜设备及原理示意图

Fig. 1 Schematic diagram and equipment of long tube deposition system for inner surface

1.2 膜层性能测试

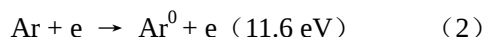
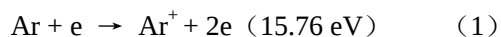
管道内壁膜层制备过程中, 采用光谱仪 (AvaSpec-ULS2048CL-4-EV0) 和光学聚焦平台对管道内 3 位置的 300~800 波段的等离子体光谱进行分析。膜层制备完成后, 采用色差仪进行膜层表面亮度测试, 确定膜层的 L^* 、 a^* 、 b^* 值, L^* 代表亮度, a^* 表示为红绿, b^* 表示为黄蓝。采用扫描电子显微镜 (SEM, Gemini 300) 观察膜层截面形貌。采用原子力显微镜 (Bruker / Dimension Icon) 测量膜层表面粗糙度。采用摩擦磨损试验机 (MS-T3001) 测试膜层耐磨性能, 选用直径 3 mm 的 GCr15 球为对磨副, 负载 3 N, 转速速度 200 r/min, 测试时间 120 min。采用纳米压痕硬度仪 (Bruker TI980) 进行膜层纳米硬度测试。采用水静态接触角测量仪 (SDC-200SH) 进行测量, 每次滴水 2 μ l, 每片试样测试三次, 计算平均值为最终测试结果。采用激光

拉曼光谱仪 (Horiba LabRam HREvolution) 对膜层进行测量, 选用 532 nm 激光光源, 主要测量范围为 800~2 000 cm⁻¹, 并采用高斯曲线对拉曼光谱进行拟合分析。

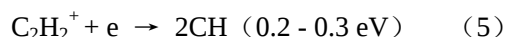
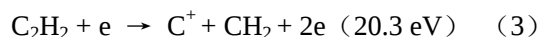
2 结果与讨论

2.1 管道内壁镀膜放电辉光光谱

在管内气体放电过程中, 管内放电区域中各粒子的光谱特性可以反映粒子的等离子体特征。基于等离子体光谱分析, 可得到工作气体离化的粒子种类、数量。在管道内壁镀膜工艺中, 第一步管内通入 Ar 气进行辉光放电清洗, 在 Ar 气放电过程中发生离化和激发^[15-16], 产生 Ar⁺和 Ar⁰, 如以下过程:



由式 (1)、(2) 可以得出, 电子能量较高时, 更有利于 Ar 的离化, Ar⁰(激发态波长大于 650 nm)、Ar⁺(电离态在 400~450 nm)^[17-18]数量增加较多, 而且 Ar⁺的增多有利于离化其他工作气体, 增强等离子体密度。工艺中乙炔被引入用来制备 DLC 膜层, 乙炔主要发生以下离化过程:



通过式 (3) 得出, 当电子能量较高时, 可以直接将乙炔离化成 C⁺; 而当电子能量较低时, 电子首先将乙炔离化成乙炔离子, 随后发生 (4) 和 (5) 过程, 生成 C₂、H 和 CH^[19], 如图 2 所示。

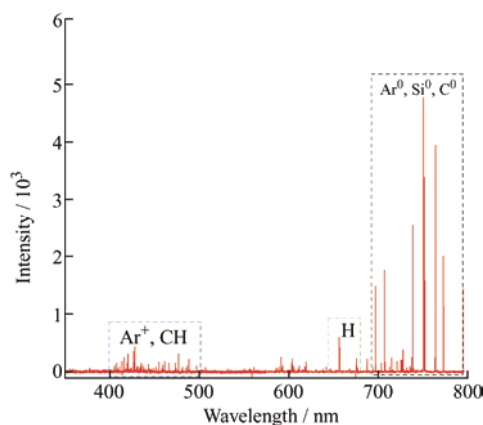


图 2 管道内 Ar+C₂H₂ 混合气氛的放电光谱
Fig. 2 Optical emission spectroscopy in the mixture of Ar+C₂H₂ in tube

2.2 管道内壁膜层截面形貌

通过 SEM 观察不同位置膜层的截面,如图 3 所示。膜层截面均匀致密,未出现裂纹、针孔等缺陷,膜层与基体结合过渡未出现裂纹。不同位置膜层厚度不同,1、3、7、9 位置的膜厚分别是 2.6、2.4、1.5、2.9 μm 。靠近管道两端的膜层厚,说明该区域的等离子体密度高,且膜层的沉积速率高。

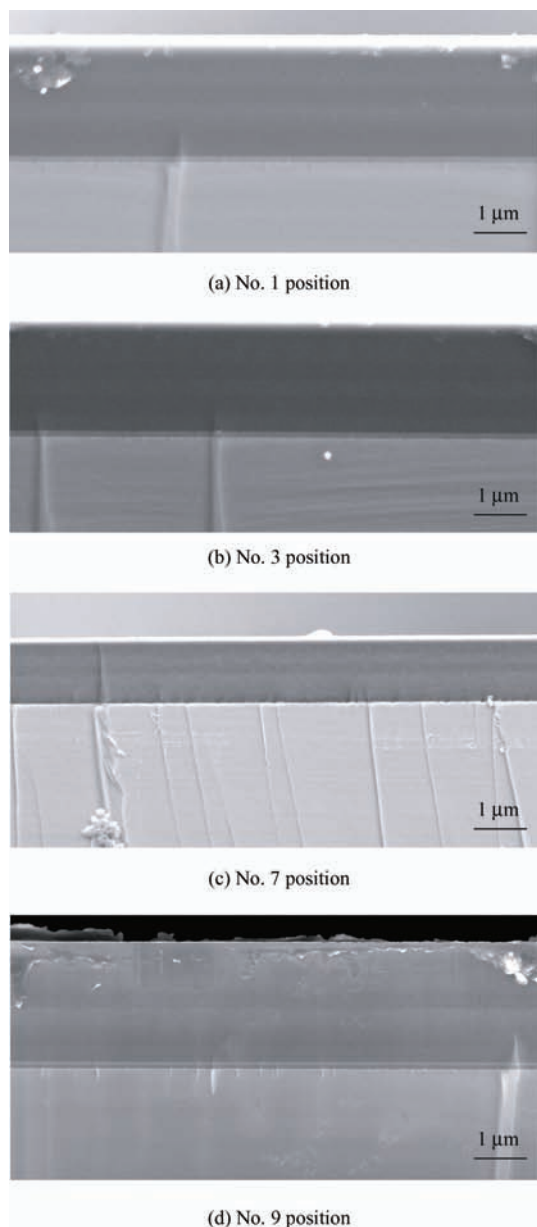


图 3 管内不同位置膜层横截面

Fig. 3 Cross sections of films at different positions inside the pipe

2.3 管道内壁膜层亮度

管道内壁膜层的均匀性直接影响着整个管道的膜层性能和管道的使用寿命,所以管道内壁膜层的均匀性十分重要。通过对试片膜层亮度的测试也可侧面反应出膜层性能的一致性。根据国际照明委员

会 (Commission Internationale De L'Eclairage, CIE) 确定的 L^* 、 a^* 、 b^* 颜色空间,两种颜色之间的总色差 ΔE^* 可以用式 (6) 计算^[20]:

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (6)$$

如图 4 所示,采用分光光度计测量膜层的表面亮度 L^* 值。可以看出图中 9 位置的膜层的亮度值最大 37.4,管道两端亮度高于中间位置的亮度。3、7、9 位置与 1 位置的色差 ΔE^* 值分别为 1.8、1.9、1.84,说明膜层亮度均匀。

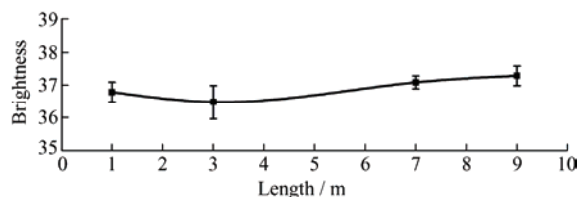
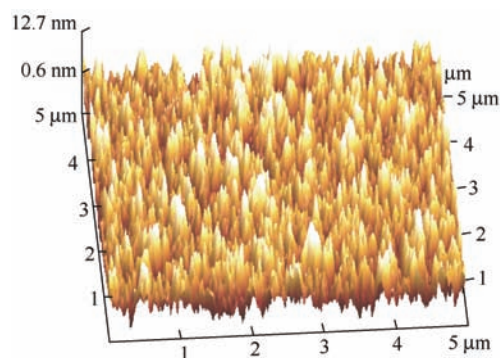


图 4 管内不同位置膜层表面 L^* 值

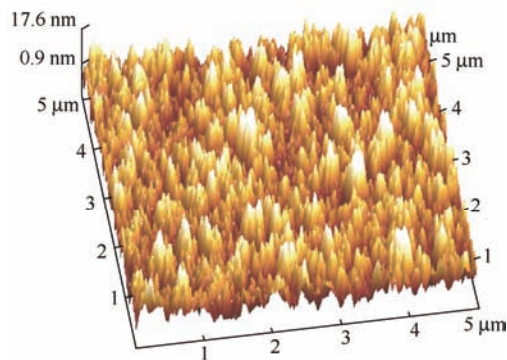
Fig. 4 Surface L^* values of films at different positions inside the tube

2.4 管道内壁膜层粗糙度

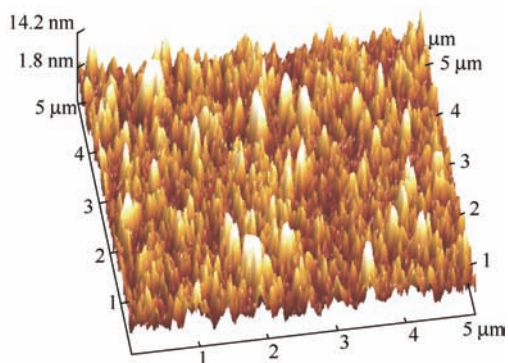
通过原子力显微镜测量膜层的表面粗糙度,以表示膜层表面的不平度。如图 5 所示,1、3、7、9 位置的膜层表面粗糙度分别是 2.82、3.91、3.25、2.58 nm。3 和 7 位置膜层表面有较多大的凸起,导致膜层表面粗糙度大;3 和 9 位置膜层表面凸起尺寸小,分布均匀。



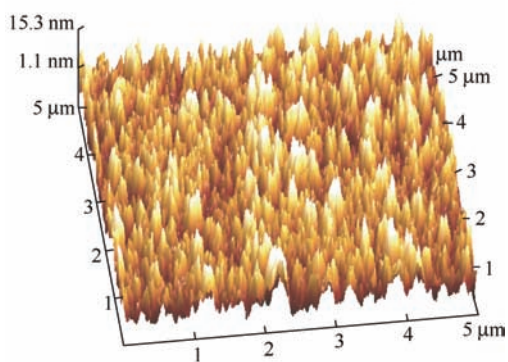
(a) Surface topography of DLC films for No. 1 position



(b) Surface topography of DLC films for No. 3 position



(c) Surface topography of DLC films for No. 7 position



(d) Surface topography of DLC films for No. 9 position

图5 不同位置膜层表面形貌

Fig. 5 Surface morphology of films at different positions

2.5 管道内壁膜层水静态接触角

管道内部一般输送的是液态物质,对管道内壁的水静态接触角有着特殊需求。如图6所示,对管道内不同位置膜层表面的水静态接触角进行测量。水静态接触角的大小一般影响因素有膜层表面的微观结构、官能团和表面能等。结果显示,在靠近管道两端(1、9位置)膜层的水静态接触角小,而靠近管道中间的膜层水静态接触角大。这可能是由于管道两端的等离子体密度高,膜层表面受到的轰击强,膜层表面较致密平滑^[21]。而中间位置的膜层表面受到轰击弱,膜层表面的微观结构使得水静态接触角增大^[22]。

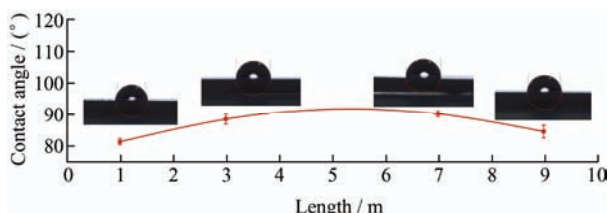


图6 管内不同位置膜层表面接触角

Fig. 6 Contact angle of films at different positions inside the pipe

2.6 管道内壁镀膜磨损测试

管道内壁沉积镀膜的一个主要目的是提高管道内壁的耐磨性能。如图7所示,对管道内3与7位置的试样膜层进行了2 h 摩擦磨损测试。结果显示,

膜层的摩擦因数在开始的 20 min 内呈减小趋势,从 0.225 降到 0.175 左右。随着时间延长,摩擦因数基本稳定在 0.17,这说明膜层在测试过程中,膜层中石墨相在摩擦磨损过程中形成转移膜^[23],为后续的磨损提供了润滑^[24-25]。对磨损后的磨痕进行观察,未发现膜层破损剥落,三维的磨痕形貌也可以看到磨痕中没有出现大的磨损坑,如图7、8所示。通过式(7)计算比磨损率 I ,即单位滑动距离和单位负载产生的体积磨损量,用于评估膜层的耐磨性。

$$I = \frac{A \cdot C}{S \cdot F_N} \quad (7)$$

式中, I 为比磨损率 (mm^3 / Nm); A 为磨损轮廓的面积 (mm^2); C 为磨痕长度 (mm); S 为滑动距离 (m); F_N 为法向载荷 (N)^[26]。得到膜层的比磨损率 I 为 $8.9 \times 10^{-7} \text{ mm}^3 / \text{Nm}$,说明膜层的耐磨性较好^[27]。

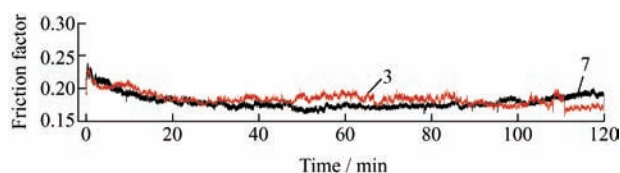
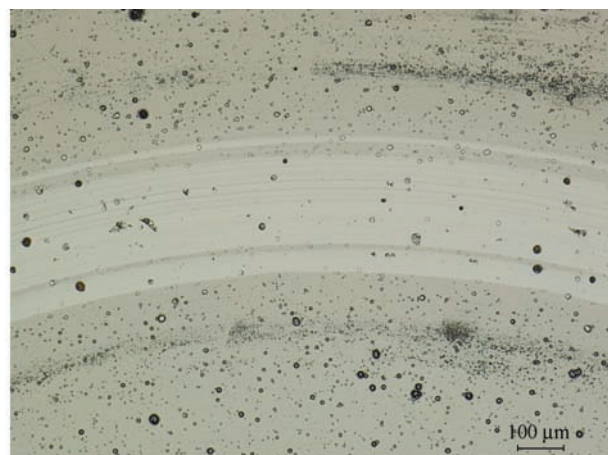
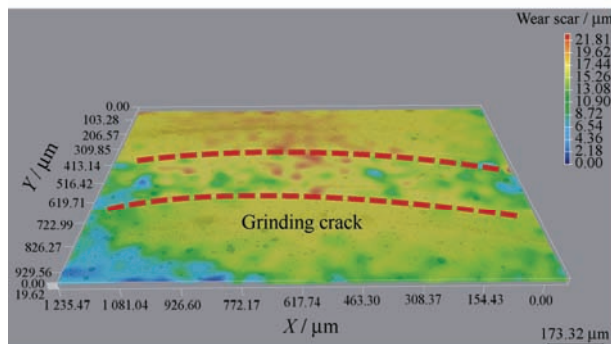


图7 3位置与7位置的膜层摩擦因数

Fig. 7 Friction factor of films at No. 3 and No. 7 positions



(a) Scratching of wearing for No. 3 position



(b) 3D morphology of wearing for No. 3 position

图8 3位置的试样磨痕及其三维形貌

Fig. 8 Scratching and 3D morphology of wearing for No. 3 position

2.7 管道内壁膜层硬度

图 9 是试样膜层的硬度与弹性模量曲线。发现膜层的硬度在管道两端较高,弹性模量也相对较高。这是因为在管道两端由于气体密度高,产生了高密度的等离子体,膜层得到强的离子轰击,使得膜层越致密,膜层的硬度也越高^[28]。在靠近管道中间位置的膜层硬度低。可能是由于管道中间位置的气体放电较弱,产生的等离子体密度低,造成沉积到薄膜表面的粒子能量低、轰击弱,从而导致制备的膜结构疏松、硬度低^[21]。

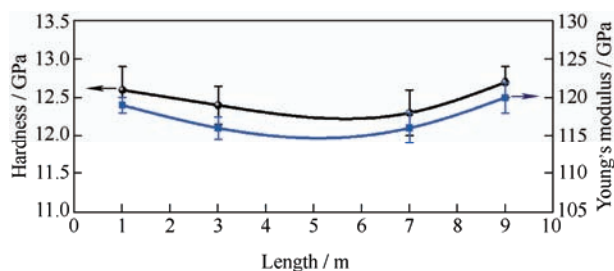
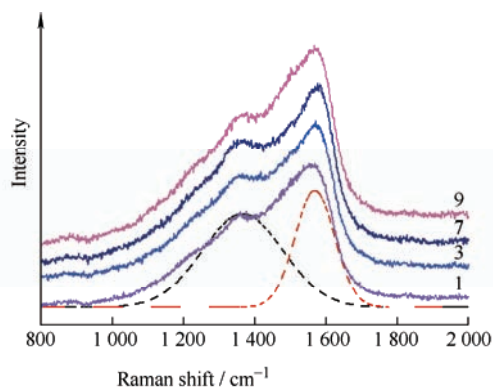


图 9 不同位置膜层硬度与模量

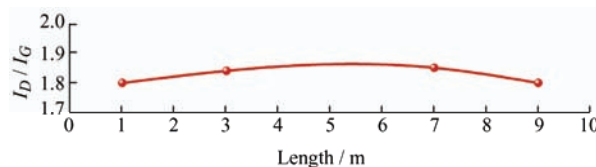
Fig. 9 Hardness and young's modulus of the film for different position

2.8 管道内壁膜层拉曼光谱分析

通过对膜层拉曼光谱进行分析,拉曼光谱被拟合合成两个高斯波段,位于 1580 cm^{-1} 附近的主峰被指定为 G 峰,在 1350 cm^{-1} 附近的被指定为 D 峰^[29-31]。膜层的 D 峰和 G 峰的强度比值 I_D/I_G 可以用于表征 DLC 膜层中非晶态结构, G 峰的位移表示膜层应力。图 10 a 所示不同位置膜层的拉曼光谱。与 1 和 9 位置对比, 3 和 7 位置 G 峰表现出向高峰位移动,说明两端的膜层应力高。图 10 b 所示膜层中 I_D/I_G 的分布趋势,管道两端的 I_D/I_G 低,说明两端的膜层中 sp^3 含量较高,且膜层硬度较高^[32-33]。



(a) Raman spectroscopy of different position



(b) I_D/I_G of different position

图 10 管内膜层拉曼光谱

Fig. 10 Raman spectra of the inner film of the tubes

3 结论

采用实验室自主研制的管道内壁镀膜设备和电源,基于空心阴极放电产生的等离子体技术,在 10 m 管道内利用 PECVD 成功制备了 DLC 薄膜,解决了在大长径比管道内壁镀膜的难题,并研究了管道内气体放电辉光光谱及 DLC 膜层性能,主要结论包括:

(1) 实现在 10 m 大长管道内部空心阴极等离子体放电,通过采集等离子体光谱,在管内发现的粒子包含 Ar^+ 、 C_2 、 CH 和激发态的 Ar^0 、 Si^0 、 C^0 。

(2) 管道内膜层的亮度和色差对比没有表现出较大差异。拉曼光谱结果表明,管道内膜层 I_D/I_G 均匀,说明在大长径比管道内壁沉积的膜层结构是均匀的,解决了在管道内部不均匀的问题。

(3) 获得 DLC 膜层硬度大于 10 GPa。管道内膜层的磨损未出现破损脱落,表现出良好的耐磨性能,延长了管道的使用寿命。

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