doi：10.3969/j.issn.1007-9289.2012.01.001

美国西南研究院等离子全方位离子镀膜技术研究及实际应用

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摘 要：文中介绍了美国西南研究院（Southwest Research Institute-SwRI）等离子全方位离子镀膜技术（PIID）的最新研究成果。SwRI从实际应用出发，在等离子表面工程领域成功研发了一些新技术，PIID技术为其中一种。近几年来，SwRI研究的重点为类金刚石（DLC）涂层的高速率沉积，制备超厚DLC涂层用于提高工件的耐磨性和耐腐蚀性能、憎水DLC涂层以及长管道内表面的镀膜技术。与传统的脉冲辉光放电（PGD）技术不同，SwRI基于空心阴极放电（HCD）等离子技术研发了一套独特的镀膜技术，同时讨论了HCD技术的原理，并对HCD和PGD技术制备DLC涂层的实验结果和这两项技术的实际应用进行了讨论。最后，对该技术最新的研究方向进行了展望。

关键词：DLC；脉冲辉光放电；空心阴极放电；管道内壁镀膜；冲蚀；磨损；腐蚀

中图分类号：TG174.442 **文献标识码：**A **文章编号：**1007-9289(2012)01-0001-10

Plasma Immersion Ion Deposition Research at SwRI and Its Practical Applications

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Abstract：This paper reviews the latest research and development at Southwest Research Institute (SwRI) in Plasma Immersion Ion Deposition (PIID). SwRI has been developing new technologies in plasma surface engineering for practical applications. One of the technologies is the PIID. In recent years，we have focused on high rate deposition of diamond-like carbon (DLC) coatings，thick DLC for improving erosion and corrosion resistance，hydrophobic DLC coatings，and the deposition of the inner surface of long pipes. A particular technique is developed based on the hollow cathode discharge (HCD) plasma process rather than the conventionally used pulsed glow discharge (PGD) process. In this paper，we will discuss the principle of the HCD process，the experimental results of the DLC coatings produced by both the PGD and the HCD processes，and practical applications of these technologies. New research directions will be presented at the end of this paper.

Key words：DLC；pulsed glow discharge；hollow cathode discharge；tube bore coatings；erosion；wear；corrosion

0 Introduction

Plasma source ion implantation (PSII) raised significant interest since the technology first appeared because of its promise of treating large 3-D components without the need of sample manipulation，thereby potentially reducing the processing cost[1-2]. Later the process has been commonly referred as plasma immersion ion implantation (PIII)[3-5]. After over two decades of effort，even though the research is still active[6-10]，PIII has not been widely

收稿日期：2011-12-17；**修回日期：**2012-01-09

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网络出版日期：2012-02-13 22:23；**网络出版地址：**http://www.cnki.net/kcms/detail/11.3905.TG.20120213.2223.011.html

引文格式：魏荣华，李灿民. 美国西南研究院等离子全方位离子镀膜技术研究及实际应用[J]. 中国表面工程，2012，25(1)：1-10.

accepted by industry for its intended tribological applications due to its inherent shortcoming, i. e. the implanted species can only penetrate to a depth no more than 200 nm. This treated layer is deemed to be too shallow for most mechanical components.

However, along with the research in PIII came another process from the late 1990s-plasma immersion ion deposition (PIID)[11-14]. Using this process, 3-D components can be deposited with a much thicker layer of hard coatings. In particular, diamond-like carbon (DLC) coating obtained using PIID has become the focal point by the research community because it can be readily deposited on a variety of surfaces. Due to its high hardness (10～25 GPa) and sufficient thickness (0. 5～2 μm), DLC has found practical applications particularly for automotive and biomedical industries[15-21]. It should be pointed out, although many techniques can be used to prepare DLC coatings including direct ion beam deposition, magnetron sputtering deposition, cathodic arc deposition, and RF plasma deposition[22-24], the pulsed glow discharge (PGD)-based PIID, a variation of plasma enhanced chemical vapor deposition (PECVD), offers a much simpler approach and hence a potential for lower processing cost[11-12, 25-28]. In addition, the deposition system hardware is also quite simple; therefore the capital investment is also low. In this paper, we will briefly discuss the PGD process; then we will focus on the latest development in the PGD process and other novel technologies. During the discussions of these technologies, we will present practical application examples.

1 Pulsed Glow Discharge Deposition

The PIID process can be understood using the schematic shown in Figure 1(a). After parts are placed in a vacuum chamber and the chamber is pumped down to a low pressure (1.3×10^{-4} Pa), a gas is fed into the chamber to reach 10～15 millitorr. When a train of pulses of negative voltage typically around 5 kV is applied to the parts, pulsed glow discharge (PGD) plasma is generated in the entire vacuum chamber. The same pulsed voltage also draws the ions out of the plasma so that they are accelerated towards the component surfaces. Depending on the ion species (precursor gases), either ion sputter cleaning (when Ar is used) or deposition of DLC (when a carbonaceous gas such as C_2H_2 is used) can be performed. These two steps are generally performed sequentially by simply switching the gas supply without breaking the vacuum. To ensure a good adhesion of the DLC to the substrate made of metallic materials, in particular steels, typically a Si or Si-C bond layer is used before the deposition of the DLC. This may be accomplished by feeding the deposition system with silane (SiH_4) or trimethylsilane (TMS). It should be pointed out that SiH_4 is pyrophoric and needs special handling, while TMS is much safer. Shown in Figure 1(b) is a cross-sectional image of scanning electron microscopy (SEM) of a DLC coating. As can be seen, the coating is dense and featureless and it shows a good adhesion to the substrate, Currently, SwRI operates one of the largest facilities (1. 2 m in diameter by 2. 5 m long) in the world. Shown in Figure 2 are a few photographs of the components that are being processed and coated with protective coatings.

2 DLC Deposition using a Mesh Method

2. 1 Principle of meshed PIID

Shown in Figure 3(a) is a schematic of the meshed PIID process[29]. Parts are installed inside a metal mesh cage. Usually the parts are electrically connected to the metal cage. When the voltage pulses are applied to the cage, plasma is generated inside the cage. The pulsed negative voltage also draws ions from all directions to the surfaces of the parts. As a result, a DLC coating can be deposited on the parts. Shown in Figure 3(b) is a photograph of the meshed PIID process. It is noted that the plasma luminescence is much stronger inside the cage than outside. In fact, this process is similar to the HCD process in tubes[30-31]. The secondary electrons generated at the component surfaces by the impact from the incoming high energy ions are trapped inside the meshed cage. These electrons, in turn, have collisions with the neutral gas and hence generate more plasma. When these electrons finally lose their energy, they will escape from the cage and go to the chamber wall to complete the circuit loop. The plasma density generated by the mesh method is much higher than that generated by conventional PGD method. As a result, a high deposition rate can be reached. Furthermore, a higher plasma density means a smaller plasma sheath; therefore, a more conformal coating can be obtained in the HCD.

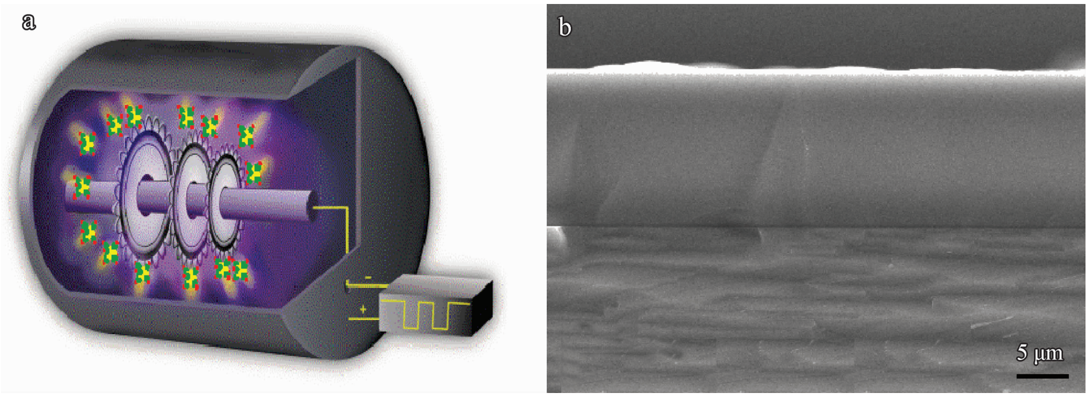

图 1 等离子全方位离子镀膜(PIID)(a)原理图(b)DLC 涂层横截面的扫描电镜图

Fig. 1 Plasma Immersion Ion Deposition (PIID) (a) Schematic (b) Cross－sectional SEM image of a DLC coating

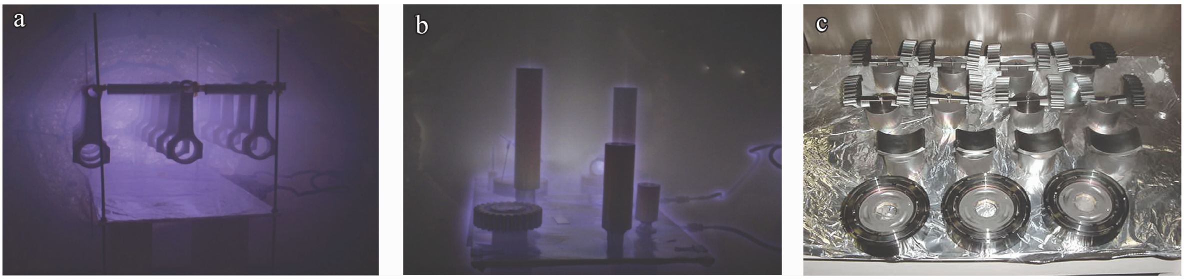

图 2 工件的 DLC 镀膜 (a)一次加工同种多个工件 (b)一次加工多种工件 (c)镀过 DLC 的工件

Fig. 2 Components coated with DLC (a) multi-components processed in one batch, (b) various components in one batch and (c) components coated with DLC

2.2　Coating uniformity, thickness and deposition rate

As shown in Figure 3(b), a number of the parts are being coated with DLC, including a tube of 58 mm in diameter by 110 mm long representing 3-D components. After the deposition, by sectioning the tube longitudinally and using SEM, the coating thickness distribution along the ID surface and the OD surface is shown in Figure 3(c). As can be seen, the coating thickness varies from 2 to 9 μm. The coating on the ID is thicker than the OD due to the hollow cathode effect even if the tube was in the meshed cage. The coating does not seem to be uniform. However, considering both surfaces can be coated simultaneously with a coating that is sufficiently thick for most applications, it may still be acceptable. In contrast, if the conventional PGD process is used to deposit the coating on both the ID and OD simultaneously, the coating coverage will be completely unacceptable. Depending on the parameters used in the process, the deposition rate of DLC can be 2～5 μm per hour for the HCD process. In comparison, the deposition rate for the PGD is only 0.5～1 μm per hr at the beginning of the deposition, while it decreases with the coating thickness. It is very hard to obtain a coating over 5 μm using the PGD process. In contrast, using the HCD process a thick coating (>20 μm) has been achieved. It should also be pointed out that using the meshed PIID method insulating components including ceramics and polymeric materials may also be coated.

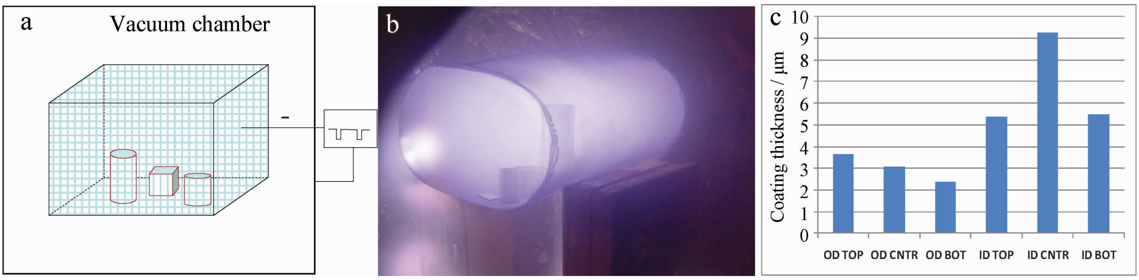

图 3 加网 PIID 技术及涂层的厚度分布(a)加网 PIID 技术的原理图(b)利用 PIID 技术正在镀膜的照片(c)管子内外 DLC 的厚度分布

Fig. 3 Meshed PIID process and the coating thickness distribution(a) schematic of meshed PIID process, (b) a photograph of the process in progress and (c) coating thickness distribution on both sides of the tube

2.3 Microstructural characteristics of DLC coatings

Quantitative Raman spectroscopic analyses on the DLC films showed that the Id/Ig is about 1.2 and 0.8 for the conventional PIID and meshed PIID, respectively, indicating an increase in the sp^3/sp^2 ratio for the DLC prepared using the meshed PIID[32-34].

2.4 Tribological characteristics of DLC coatings

Ball-on-disc tribotests were conducted on 1018 carbon steel deposited with DLC films using both methods in ambient environment, dry sliding with 1 N load against an alumina ball. Shown in Figure 4(a) are the coefficients of friction (COF) of the DLC films. Both DLC films show much lower COF than the uncoated 1018 steel (not shown, $\mu = 0.6 \sim 0.7$), while the DLC prepared using the meshed PIID shows slightly higher COF ($\mu = 0.12$) than the PGD DLC, which may be the result of a higher sp^3 content in the meshed PIID.

DLC films may be used for the protection of components under two-phase flow situations such as air and sand. The erosion resistance of the DLC films was determined using a micro sand blaster with alumina powder of an average grain size of 50 μm at an incident angle of 15° and a back pressure of 34.475 kPa. Shown in Figure 4(b) is the mass loss for selected DLC films prepared using the PGD and the meshed-PIID methods. The total erosion time was 2 min. The mass loss of the PGD DLC is slightly lower than that for the uncoated steel but the mass loss for the meshed-DLC decreases further indicating that a thicker DLC coating can provide a more durable erosion resistance.

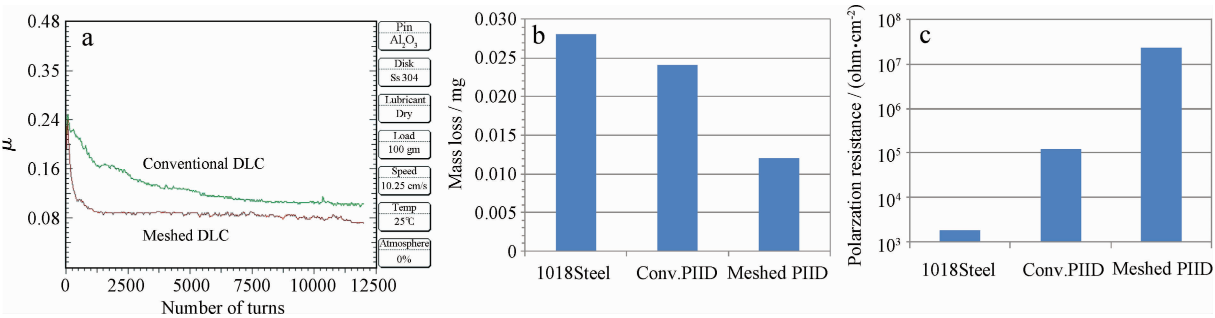

图 4 1018 钢、标准 DLC 和加网 DLC 涂层的摩擦学和耐腐蚀性能 (a)摩擦因数 (b)耐冲蚀磨损性能(c)耐腐蚀性能

Fig. 4 Tribological characteristics and corrosion resistance of 1018 steel, standard DLC and meshed-PIID DLC coatings (a) coefficient of friction, (b) erosion resistanc and (c) corrosion resistance

2.5 Corrosion resistance

Corrosion resistance of the deposited samples was evaluated using electrochemical impedance spectroscopy. Equivalent circuit models were used to calculate the polarization and pore resistance values. The results are shown in Figure 4(c). Higher polarization resistance implies better corrosion resistance. As can be seen, the DLC deposited using the meshed PIID method has much higher corrosion resistance than the standard DLC and the uncoated steel. An increase of 4 orders of magnitude is obtained. One of the reasons may be that the meshed PIID DLC is quite thick.

3 DLC Deposition on the Inner Diameter of Tubes and Pipes

3.1 Deposition of short tubes

Besides the 3-D processing capability, another advantage of the PIID technology over physical vapor deposition (PVD) processes lies in that the inner surface of a tubular structure can be deposited fairly easily. For typical size of tubes (20～100 mm in diameter by 0.1～3 m long), the ID can be deposited with DLC using various methods including hollow cathode discharge[35-37]. If the ID is very small and the material is paramagnetic, magnetic field may be used to generate plasma inside the tube[31]. Because DLC coatings are hard and chemically inert, they may be deposited on the ID of tubes for transporting crude oil or natural gas to reduce wear and corrosion from the media with small amounts of sand and water. They may also be used for tubes in steam turbines in which the two-phase flow (water droplets and steam) causes serious erosion and corrosion. This technique may also be applied to the cylinder liner bores of automotives to reduce wear.

Shown in Figure 5(a) is a photograph of a short tube with a small diameter (25 mm), while Figure 5(b) two short tubes with a larger diameter (100 mm) in a DLC deposition process. In general the smaller the diameter, the higher the pressure is needed to generate plasma inside the tube. For a tube of 25 mm in diameter by 300～400 mm long, the breakdown pressure is about 50 millitorr either for Ar or acetylene.

3.2 Deposition of long pipes

Long pipe may also be deposited with DLC films. Shown in Figure 6 is a large diameter (150 mm), long curved carbon steel pipe (2 m) being deposited with DLC inside the SwRI large PIID chamber. If a pipe is too long, no vacuum chamber can be used to host the pipe. In this case, both ends of the pipe are connected to vacuum chambers with pumps, so that the pipe serves as part of the vacuum system. The pipe can be electrically isolated from the vacuum chambers using tubular insulators. By applying high voltage pulses to the pipe, hollow cathode discharge can be generated inside the pipe and a DLC film can be deposited on the inner surface. Shown in Figure 7 is a photograph of the deposition system, using which long pipes over 10 m can be deposited.

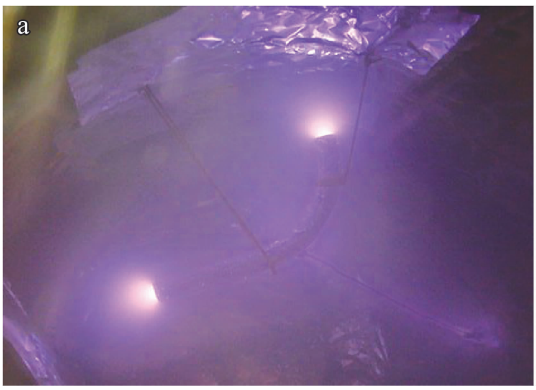

图 5 使用空心阴极放电 PIID 技术细管和管道内壁沉积 DLC (a) 一根细弯管 (b) 两根短粗管

Fig. 5 Deposition of short tube and long pipes using HCD PIID process (a) one short tube with a small ID (b) two short tubes with large ID

图 6 使用空心阴极放电 PIID 技术对长管道内径镀 DLC 膜

Fig. 6 Deposition of DLC on ID of long pipe using HCD PIID process

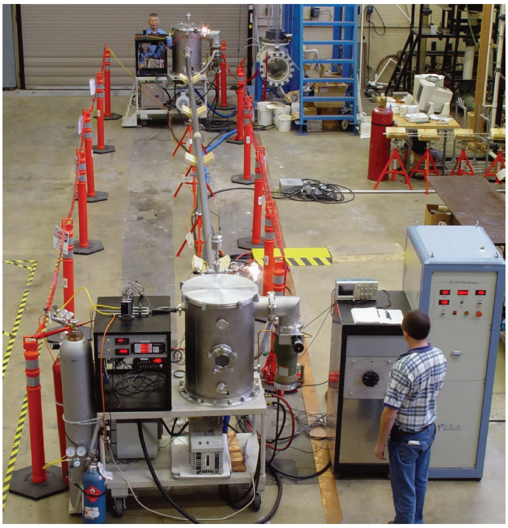

图 7 长管道的镀膜系统

Fig. 7 Deposition system for long pipes

3.3 Morphology of DLC in long pipes

To study the DLC coating morphology, a long pipe was sectioned and the samples were studied using SEM. Shown in Figures 8(a) and 8(b) are the low and high magnifications of cross-sectional SEM images of the DLC coating on the steel pipe surface. In this deposition a Si-C bond layer was used first; then the DLC coating was deposited on top. As can be seen, the coating is quite conformal. Even though the steel pipe surface is very rough, the DLC coating covers everywhere and adheres well to the substrate. Certainly, our systematic studies indicate that, in some cases, if the substrate surface is too rough with deep cuts or kinks, the DLC film will have discontinuities. The discontinuities or cracks in the coating are detrimental to the corrosion resistance. Therefore, it is necessary to polish the surface to reduce the surface roughness and hence to reduce the number of defects.

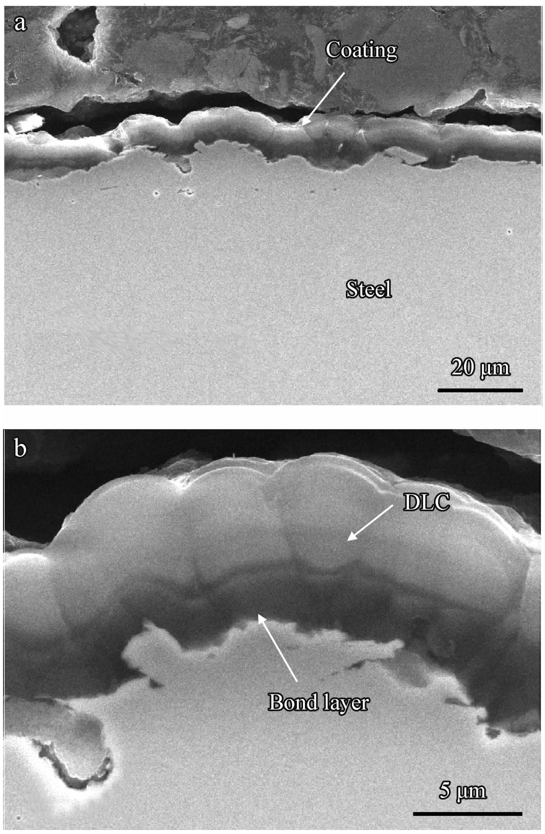

图 8 长管道内径镀 DLC 膜的横截面扫描电镜图 (a)低倍(b)高倍

Fig. 8 Cross-sectional SEM views of DLC deposited long pipe ID (a) low magnification and (b) high magnification

4 DLC Coating Application Examples

4.1 Hydrophobic DLC coatings

Surface energy of a material surface is a physical property that can be characterized by water contact angle (WCA). It is defined that when the WCA is <90°, the surface energy is high or the surface is hydrophilic. When the WCA is >90°, the surface is low or the surface is hydrophobic. If the WCA is >150°, it is super hydrophobic. A classic example of a super hydrophobic surface from nature is the lotus leaf. Research has revealed that surface texturing is very important to achieve high WCA. In fact, lotus leafs are naturally "textured" with both micro- and nano-sized features that can hold air to achieve super hydrophobicity. Surfaces with super hydrophobic properties have many applications. The self-cleaning nature of super hydrophobic surfaces has been used for mirrors and lenses in telescopes and solar arrays to avoid dust accumulation. A hydrophobic surface may also be ice-phobic and, therefore, may be used for deicing of aircraft wings or high voltage transmission lines.

DLC coatings can be used to reduce surface energy. Shown in Figure 9 is the WCA measured in air for Al treated with different processes. The WCA for as-received (AR) aluminum sample (fairly smooth) is 83°. The "standard" DLC film deposited using C_2H_2 is slightly hydrophobic with a WCA of 93°. Since the PIID process is a PECVD process, during the deposition of a DLC film, many other gases containing elements such as F and Si can be included to form F-DLC or Si-DLC. The F-DLC deposited using C_4F_8 has a WCA of 117°. To increase the durability, a DLC can be deposited on the Al substrate; then the F-DLC can be deposited on top. The WCA of the bi-layer coating is 119°, nearly identical to that without the DLC base coating. The WCA for textured surfaces using simple sand blasting is also shown in Figure 9. Except for the uncoated samples, the WCA has increased significantly for all treatments. It is understood that even though more sophisticated surface texturing may further increase the WCA, from engineering point of view, sand blasting can be performed easily and the effect is significant. In addition to the F-DLC, Si-DLC and Si-O-DLC coatings are also used to reduce the surface energy.

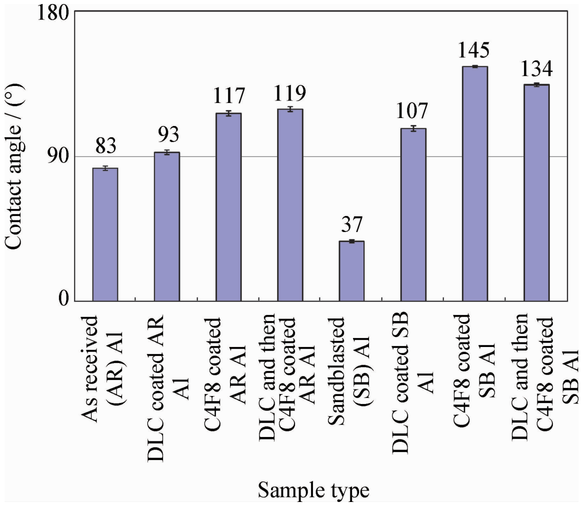

图 9 未处理(AS)和喷砂(SB)后的 Al 表面镀各种 DLC 膜的水接触角

Fig. 9 Water Contact Angle of as-received (AS) and sand-blasted (SB) Al surface coatinged with various DLC films

4.2 Deposition of large components and large number of components

In an early publication, the PIID process was demonstrated for depositing DLC on a large number of pistons[37-38]. At SwRI, the deposition of large components or a large number of small components are pursued which may be found in Ref[39]. In the following, a few more examples will be given.

Shown in Figure 10(a) is a large impeller being deposited with a DLC coating at the SwRI's PIID

图 10 大型工件和大批量工件的镀膜 (a)西南研究院大型叶轮的镀膜(b)永信等离子科技在网内同时镀三个叶轮(c)永信等离子科技一次镀 10 个潜水泵部件

Fig. 10 Deposition of large components and large number of components (a) one large impeller at SwRI, (b) three impellers in mesh at Yongxin Plasma Technology and (c) ten large water pump parts at Yongxin Plasma Technology system. The intended use for the impeller is for air compression in chemical plants in which corrosion and erosion occur to the impellers. In addition, dusty chemical particles may also adhere to the surfaces of the impellers. In the worst scenario, the buildup may eventually plug the air channels. For this application, DLC coating may be the best, or the only option, because not only is the DLC coating corrosion resistant, erosion resistant and hydrophobic, but also it can be deposited inside the narrow channels as well as the outside. Shown in Figure 10(b) is a picture of three impellers being processed using the meshed PIID process at the Yongxin Plasma Technology, Hefei, China. SwRI has helped Yongxin and established a large DLC deposition system in Hefei. Shown in Figure 10(c) are 10 large parts for water pumps being processed in the Yongxin DLC deposition system.

Shown in Figure 11 are a few photographs showing that a large number of parts are being coated or already coated with DLC in a single batch using the PIID process.

图 11 使用 PIID 工艺正在镀或已经镀过 DLC 膜的大量工件 (a) 大批密封圈的 DLC 镀膜 (b)(c) 汽车部件和试样的 DLC 镀膜 (d) 纺织机梭子的 DLC 镀膜

Fig. 11 A large number of parts are being coated or already coated with DLC in a single batch using the PIID process. (a) DLC deposition of large number of seals, (b) and (c) DLC deposited automotive components and test samples, and (d) DLC deposited shuttles for textile production

5 Conclusion and Outlook

In this paper, we presented an overview on recent research and development of the plasma immersion ion deposition process and the diamond-like carbon coatings at SwRI. A hollow cathode discharge based mesh PIID was emphasized. The coating wear resistance, coefficient of friction, erosion resistance and corrosion resistance were studied and found to be comparable to those from conventional PIID. The major advantages of this method over the conventional PIID method include the high deposition rate (up to 6.5 times), capability for thick coating (up to 20 μm) and much conformal coating coverage. Beside the "standard" DLC coatings derived from a carbonaceous gas such as C_2H_2, other

DLC coatings including the hydrophobic DLC coatings derived from Si-DLC F-DLC and Si-O-DLC were also discussed. In addition, we presented the technique on the deposition of tubes and pipes of various shapes and sizes. The PIID process may be the easiest way to the coating of ID of tubes and pipes. Up to 12 m long pipes have been deposited with a fairly uniform coating. Finally, the deposition of large components or a large number of components was also discussed with actual application examples.

For the future research, first, the development of coatings with super hydrophobic properties (WCA>150°) will find many practical applications. Second, deposition of longer pipes is challenging and needs more work. Third, high rate deposition is an important research area. Fourth, the optimal packing density in a processing chamber needs to be studied. Finally, as it has been, increasing the coating adhesion is an everlasting research topic. As the new technologies are being developed, it is believed that the PIID process will find more applications in industry.

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·学术动态·

“焊接与再制造”国际论坛将在北京召开

2012 年“焊接与再制造”国际论坛将于 6 月 1 日至 6 月 3 日在北京召开。本次论坛由中国机械工程学会及其焊接分会、再制造分会、北京工业大学以及再制造技术国家重点实验室主办，以“再制造工程中的焊接技术”为主题，目的是在“循环经济”的理念引导下，将焊接技术的特点与“再制造”相结合，加快建设资源节约、环境友好的新型制造产业，为促进包括焊接行业在内的我国工业发展方式的转变提供一个新的技术途径。

（常青 供稿）