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Micro/Nanomechanical and Tribological Properties of Thin Diamond-Like Carbon Coatings

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Abstract: The diamond-like carbon (DLC) films with different thicknesses on 9Cr18 bearing steels were prepared using vacuum magnetic-filtering arc plasma deposition. Vickers indentation, nanoindentation and nanoscratch tests were used to characterize the DLC films with a wide range of applied loads. Mechanical and tribological behaviors of these submicron films were investigated and interpreted. The hardnesses of 9Cr18 and DLC, determined by nanoindentation, are approximately 8GPa and 60GPa respectively; their elastic moduli are approximately 250GPa and 600GPa respectively. The friction coefficients of 9Cr18, DLC, organic coating, determined by nanoscratch, are approximately 0.35, 0.20 and 0.13 respectively. It is demonstrated that nanoindentation and nanoscratch tests can provide more information about the near-surface elastic-plastic deformation, friction and wear properties. The correlation of mechanical properties and scratch resistance of DLC films on 9Cr18 steels can provide an assessment for the load-carrying capacity and wear resistance.

Key words: diamond-like carbon; microhardness; nanoindentation; nanoscratch; solid lubricating

类金刚石薄膜的微/纳米机械和摩擦性能．张泰华，刘东旭，郇勇，杨业敏，王秀兰．中国航空学报（英文版），2003，16(1)：47－51．

摘　要：采用真空磁过滤等离子电弧沉积的方法在 9Cr18 **钢上沉积不同厚度的** DLC **膜。为了检测成膜质量，在较宽的载荷范围内分别使用显微硬度、纳米压痕和划痕技术表征** 9Cr18 **钢和** DLC/9Cr18 **的机械和摩擦性能。结果显示，**9Cr18 **和** DLC **的纳米硬度和弹性模量分别为** 8GPa、250GPa **和** 60GPa、600GPa，9Cr18、DLC **和有机膜的摩擦系数分别为** 0.35、0.20 **和** 0.13。**纳米压痕和划痕技术能为** DLC/9Cr18 **提供丰富的近表面弹塑性变形和摩擦、磨损等信息。**DLC/9Cr18 **的机械和摩擦性能的研究可以用来评估膜的承载和抗磨损性能。**

关键词：类金刚石膜；显微硬度；纳米压痕；纳米划痕；固体润滑

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Diamond-like carbon (DLC) materials are amorphous forms of carbon with excellent frictional properties[1,2] and high hardness[3]. DLC overcoats have been used as wear protective overcoats on engine parts, tools, hard disks and magnetic recording head to extend their working lifetime[4]. Bull and Chalker[5] have reviewed a number of applications of DLC coatings. These applications require a detailed understanding of the mechanical and tribological properties of DLC films on substrates. The properties of DLC films vary with deposition conditions. Therefore, a thorough understanding of the mechanical response of a coated component to applied loads is of both fundamental and technical importance to evaluate the surface hardening and interfacial bonding strength for films on substrates.

Since the mechanical properties affect the friction and wear performance, the study of the mechanical properties of DLC thin coatings is of primary concern. The correlation of mechanical properties and scratch resistance of films on substrates

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can provide an assessment for the film quality and wear effect. Therefore, the present study seeks a direct quantitative evaluation of the tribological properties of DLC films on 9Cr18 steels by using microhardness, nanoindentation and nanoscratch techniques.

1 Experimental Details

1.1 Film preparation

The substrate material was 9Cr18 bearing steel (with composition in wt. %: Si-0.8, Mn-0.72, P-0.035, S-0.03, C-0.96, Cr-17.8, Fe-79.655). The specimens were cut from real bearing rings that had been quenched-and-tempered. Their dimensions were 25mm in outer diameter, 10mm in inner diameter and 3mm in thickness. They were mechanically polished to a surface roughness, R_a, of 0.02μm. Before deposition, the specimens underwent ultrasonic cleaning progressively in gasoline, acetone and ligroin.

Deposition of the DLC films was carried out by a vacuum magnetic-filtering arc plasma method. The steel specimens were put into the vacuum chamber, which was pumped until the base pressure of 3×10^{-3}Pa was achieved, and then they were cleaned by the plasma beam bombarding. A bias voltage was added on the samples. By controlling the deposition rate and time, the DLC film thickness of about 0.3μm and 0.5μm can be deduced. Finally, the DJB823 organic coating of approximately 1.0μm in thickness was deposited on the DLC films.

1.2 Mechanical characterization

Surface observation was performed by a POLYVAR MET® optical microscope, coupled with a Vickers microhardness instrument. HV tests were performed using the following conditions: load 200g, holding time 30s.

Nanoindentation and Nanoscratch tests were conducted using an MTS Nano Indenter® XP with a Berkovich diamond tip[6,7]. The hardness and elastic modulus were measured using the continuous stiffness measurement (CSM) option. A fused silica was used as a standard sample for the initial calibration. In the present study, the experimental parameters were chosen as follows: strain rate, 0.05s^{-1}; allowable thermal drift rate, 0.05nm/s; depth limit, 1.0μm. Five load-displacement curves were recorded for each sample. The hardness and elastic modulus were obtained using the Oliver-Pharr method[8]. All tests were carried out at room temperature.

Scratch tests were performed with LFM option of the Nano Indenter® XP. Berkovich tip was used with face forward. During a scratch test, the ramping normal force on the indenter was held. For these tests, the maximum scratch loads were 40mN, 100mN and 300mN respectively. A scratch velocity of 10μm/s was used by controlling the stage movement. A typical scratch experiment is performed in nine subsequent steps: approaching the surface; first profile with normal load 20μN for 700μm in the *Y* direction; return profile; pre-profile for 100μm; scratch profile with ramping normal load for 500μm; unload; post-profile for 100μm; return profile; final profile.

2 Results and Discussion

2.1 Microhardness

The surface mechanical properties of 9Cr18 steels and DLC/9Cr18 were evaluated by microhardness indentation technique. The values of Vickers microhardness were calculated from five indentations for each sample. Typical indentation micrographs are shown in Fig. 1. The indentation of 9Cr18 is well marked. Here is the experimental result of $\text{HV}_{0.2}=682.88\pm24.77$. For DLC/9Cr18, the indentation edge was too blurry to measure the indentation area exactly in Fig. 1. Therefore, it could not give exact microhardness values.

2.2 Nanoindentation properties

In general, microhardness data represent composite effect from various components, since the indented volume was extended over regions of different components, as shown in Fig. 1. With the nanoindentation technique, it is now possible to determine the hardness and elastic modulus of

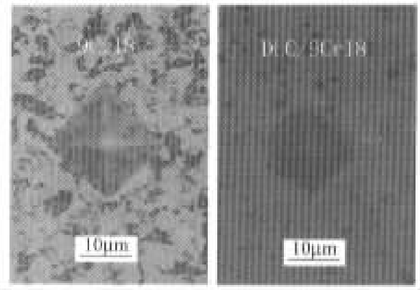

Fig. 1 Optical micrographs of 9Cr18 steel and DLC/9Cr18

thin films in the near-surface range, for example on the scales from microns to nanometers[6-8]. These properties are derived from the load-displacement curves. The average hardness and modulus of 1000nm depth are approximately 8GPa and 250GPa respectively. The dimensions of the components for 9Cr18 steels are essential in micron scale, as shown in Fig. 1. The nanohardness data are more dispersive than the microhardness ones. This may be ascribed to the nanoindentation performed on the different components.

The load, hardness and modulus *vs* displacement curves for 9Cr18-DLC/9Cr18 with the thickness of about 0.3μm and 0.5μm are shown in Fig. 2, respectively. Nanoindentation tests showed that the hardness and modulus values of 9Cr18 steels were essentially independent of depth, and the values of DLC/9Cr18 were dependent on depth. When indentation depths were below 20% of the film thickness, the substrate effect on hardness and modulus can be ignored[9]. Variations in surface topography led to the scatter in measured values within 50nm of the film surface[10]. Therefore, hardness and modulus values for DLC films were displayed approximately 60GPa and 600GPa from 50nm to 100nm, roughly less than one-fifth of DLC film thickness in order to avoid the influence of the substrates. With the depth increasing, hardness and modulus gradually decrease, which can be attributed to the contribution from DLC coatings and steel substrates. When the depths reached 1000nm, the contribution of underlying substrates became more pronounced, and the hardness and modulus values were close to those of the substrates. In Fig. 2(a), unloading 90% of the peak load, the residual indent depths for DLC/9Cr18 were shallower than for 9Cr18 steels. So, DLC film exhibits a better elastic recovery capacity.

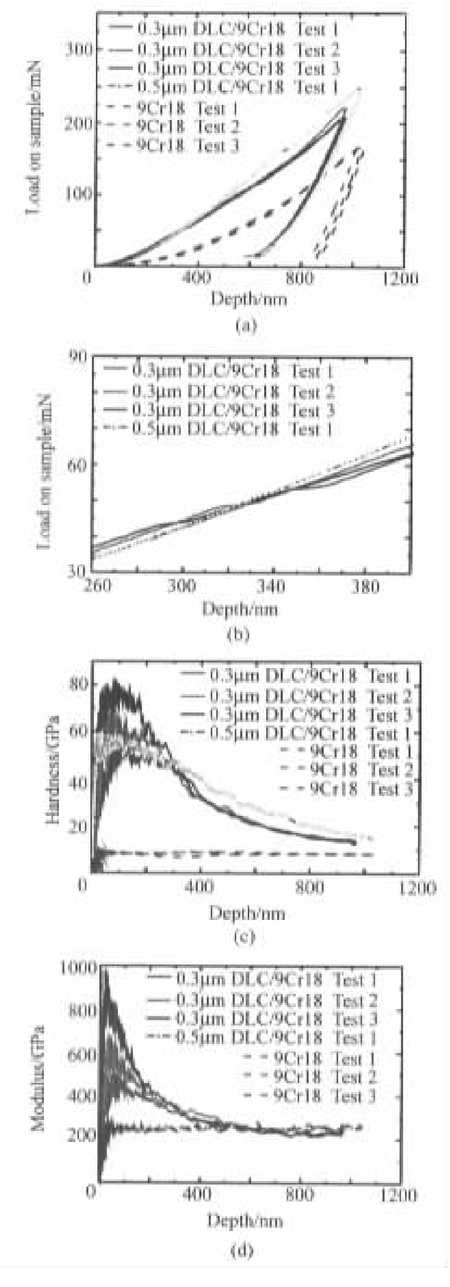

Fig. 2 Load on sample, hardness and modulus as a function of depth for different samples (For plotting clearly, the first three indent results of five indentations were given)

The discontinuities in loading curves which can be clearly seen in Fig. 2(a) and Fig. 2(b) were reproducible and were indicative of adhesion failure. Using the indentation approach, it was clearly shown that the DLC adhesion was strongly dependent on the particular deposition technique employed.

2.3 Nanoscratch properties

In a scratch test, the cracking or delamination of a hard coating is marked by a sudden increase in the friction coefficient curve[9]. The normal load associated with this event is called the critical load. The friction coefficient, normal load and lateral load as a function of *Y* displacement and optical images made on the organic/DLC/9Cr18 and the DLC films are shown in Figs. 3-5.

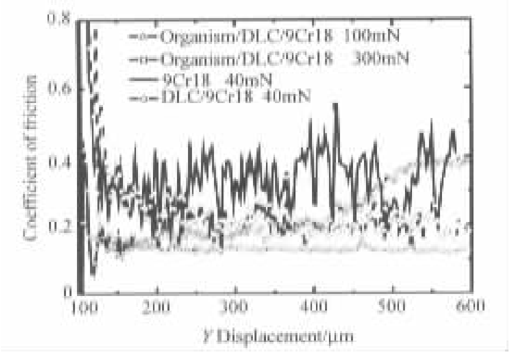

Fig. 3 Friction coefficient as a function of *Y* displacement for 9Cr18 steel and DLC/9Cr18 and organic/DLC/9Cr18 at different loads

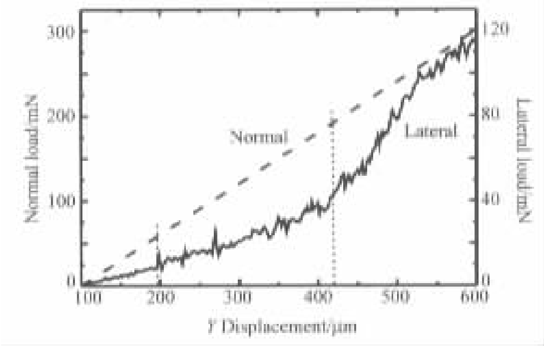

Fig. 4 Normal load and lateral load as a function of *Y* displacement for organic/DLC/9Cr18 at normal load 300mN for about 1.0μm thick organic coatings and about 0.3μm thick DLC coatings

Five nanoscratch tests were run on each specimen in order to study the property of wear resistance. At a peak normal load of 40mN, 9Cr18 steel exhibited a friction coefficient of 0.35 and big variations. This may be attributed to the different tribological properties from various components of steels, as shown in Fig. 1(a). The DLC friction coefficient for a peak normal load of 40mN exhibited smaller variation, namely about 0.20 in Fig. 3, showing that the solid lubricating effect of DLC films is very marked.

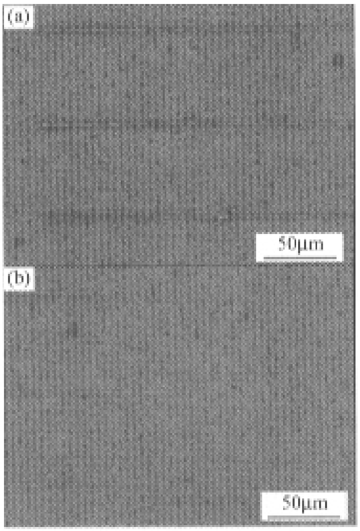

Fig. 5 Optical micrographs of residual scratches for organic/DLC/9Cr18 with the ramping normal force from top (20μN, 100μm) to bottom (300mN, 600μm) over a scratch distance of 500μm and the top scratch corresponding to the 300mN curves in Fig. 3

The organic films with the thickness of about 1.0 m were coated on DLC films. In Fig. 3, the friction coefficient of 0.13 for organic/DLC/9Cr18 at a peak normal load 100mN was lower than that of DLC/9Cr18 at 40mN, exhibiting that the lubricating effect of the organic film was improved obviously. The friction coefficient curve at a peak load of 300mN for 9Cr18 was divided into three stages: Firstly, it kept no change between 100μm and near 200μm, showing that it was the same as that at 100mN. Here the tip scratched in the organic film, so the lateral load curve smoothed in Fig. 4. Secondly, the friction curve rose gradually from 200μm to 420μm and exhibited a big variation, showing the tip scratching into the DLC coatings. This may be because of the occurrence of nonuniform plastic deformation and cracking in the DLC coatings. In Fig. 4, the related normal load and lateral load at Y displacement 200μm are 57.7mN and 8.7mN, respectively, namely the critical load of the organic film. Finally, the friction coefficient curve sharply increased between 420μm and 600μm, showing the tip scratching into 9Cr18 steel. In Fig. 4, the related normal load and lateral load at Y displacement 420μm are 192.2mN and 42.6mN, respectively, namely the critical load of DLC film. It is observed that the very small debris was on the sides of scratch in Fig. 5 top scratch. The debris would result in premature failure in bearings. The small debris for organic/DLC/9Cr18 peeled off around the scratch. The failure mode of DLC film is mainly brittle fracture.

3 Conclusions

This paper studied the micro/nanomechanical and tribological properties of the solid lubricating DLC films on 9Cr18 steels. The main conclusions may be summarized as follows:

The hardnesses of 9Cr18 and DLC, determined by nanoindentation, are approximately 8GPa and 60GPa respectively, and their elastic moduli are approximately 250GPa and 600GPa respectively. The friction coefficients of 9Cr18, DLC, organic coating, determined by nanoscratch, are approximately 0.35, 0.20 and 0.13 respectively. So, compared with 9Cr18, the mechanical and wear resistance properties of DLC/9Cr18 and organic/DLC/9Cr18 are significantly improved.

The modulus and hardness of substrates should approach those of DLC films when choosing substrate materials. This may be due to the close properties between films and substrates, which can reduce the internal stress field and improve the critical load.

The nanoindentation and nanoscratch tests can provide very useful information on the near-surface elastic-plastic deformation, fracture, friction and wear properties.

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