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Thesis Title: Deposition and characterization of non-uniform structure adamant films

Adamant film is one of the promising surfaces protective coating can be applied in various engineering fields. This dissertation will focus on the adamant film, which consists of boron, carbon and hydrogen elements. The main objective of this study is to improve the mechanical, tribological and biocompatibility of adamant films by surface design and boron incorporation. First attempts, will be made to produce segment-structure on hydrogen-free diamond like carbon (DLC) by filtered cathodic vacuum arc (FCVA) method. Next attempts will focus the deposition of porous boron carbide film from trimethylboron (TMB) by pulsed plasma chemical vapor deposition (CVD). This section will provide a new sight in the boron carbide deposition which contain pores which is to be found later effective to be used as surface protecting coating with excellent tribological behavior. The potential of non-uniform and uniform structure surface of boron carbide film with different boron content to be used as coatings in blood contacting device will be also explored.

This thesis is divided into six chapters; Chapter 1 is an overview of adamant films. Briefly introduced the terminology used in the thesis and related background on non-uniform surface structure of carbons. Non-uniform structure will be introduced on the surface of adamant films with the depth scale of 1 to 600 nm.

In Chapter 2, "Deposition and mechanical characterization of segment-structured hydrogen-free DLC films by filtered cathodic vacuum arc (FCVA) method," has demonstrated the work for obtaining segment structure using metal meshes as masks on hydrogen free DLC film. It was found that segment-structured hydrogen-free DLC films ($220 \times 220 \mu\text{m}^2$ of segment with $30 \mu\text{m}$ of groove width) have approximately 25% less internal stress than a continuous film without changing their mechanical properties. The maximum film thicknesses were 500 and 600 nm for silicon and SUS403 substrates, respectively owing to a limitation imposed by high compressive stress. Tribological test results indicate that the segment-structured hydrogen-free DLC films have excellent wear resistance and a lower friction coefficient than conventional hydrogen-free DLC films. The role of grooves in reducing internal stress and trapping the wear debris explains this better performance. Furthermore, a thicker segment-structured

hydrogen-free DLC film can suppress the intensive wear of the segment edge. Overall results in this chapter, indicate the segment-structured hydrogen-free DLC films have the advantages of reduced stress and improved the tribological performance.

In Chapter 3, “Deposition of amorphous boron carbide (*a*-BC:H) films for trimethylboron (TMB),” discussed the characteristic of porous *a*-BC:H film as an alternative for segmented DLC films in application where severe tribological conditions and complex shape exist. Several depositions were done by varying deposition parameter such as pressure and TMB gas flow rate in order to obtain porous film surfaces by pulsed plasma chemical vapor deposition (CVD). Their mechanical and tribological behavior will be compared in order to obtain the optimum condition for the deposition. A local oxidation experiment was carried out in humid condition for 90 h on this film in order to study the pores stability. The results show that *a*-BC:H films which deposited with higher TMB flow rate consist of higher boron content and exhibits more pores. As a summary, the optimum condition deposition of *a*-BC:H film was at 5 Pa of gas pressure with 15 cm³/min of TMB gas flow rate. This film with is 0.43 B/C ratio shows the good quality characteristic of film. 0.43 B/C ratio film exhibits lower friction coefficient (0.2) sliding against stainless steel ball, high hardness (8.1 GPa) and Young’s modulus (62.2 GPa). From the humidity test done on the porous *a*-BC:H films it is observed that humidity diffusion at the pores will spread through the film thickness, finally reaching the film-substrate interface, and cause film delamination. This delamination can be unpredictable and cause early life failures. In order to prevent this delamination mechanism, DLC film was applied between the *a*-BC:H film and substrates in subsequent experiments in this study.

In Chapter 4, “Characterization of porous amorphous boron carbide (*a*-BC:H) films,” The thermal stability and tribological behavior of nano-porous *a*-BC:H films are studied and compared with those in conventional DLC films. Thermal stability of *a*-BC:H films, with no delamination signs after annealed at 500°C during 1 hour, is better than that of the DLC films, which completely disappeared under the same conditions. Tribological test results indicate that the *a*-BC:H films, even with lower nano-indentation hardness than the DLC films, show an excellent boundary oil lubricated behavior, with lower friction coefficient and reduce the wear rate of counter materials than those on the DLC film. The good materials properties such as low modulus of elasticity and the formation of micro-pores from the original nano-pores during boundary regimes explain this better performance. Results show that porous *a*-BC:H films may be an alternative for segmented DLC films in applications where severe tribological conditions and complex shapes exist, so surface patterning is unfeasible.

In Chapter 5, “Deposition and characterization of amorphous boron incorporated DLC films,” *a*-BC:H films with different boron/carbon (B/C) ratio were fabricated by pulsed plasma CVD. *a*-BC:H films with different B/C ratios (0 to 0.4) were prepared by controlling the flow rate of TMB in the reaction gas mixture with C₂H₂. *a*-BC:H film with the low B/C ratio of 0.03 exhibited the highest nano-indentation hardness (10.9 GPa) as well as the lowest friction coefficient ($\mu = 0.1$) and wear rate (3.2×10^{-7} mm³/Nm). The *a*-BC:H films with B/C = 0.03 and 0.4 exhibited high-hydrophilicity surfaces owing to their high wettability and high surface energy. An *in vitro* platelet adhesion experiment was conducted to clarify the effect of boron on the blood compatibility properties of *a*-BC:H films compared with those of Ti and TiNb substrates as well as conventional DLC. The results showed that DLC and *a*-BC:H films can reduce platelet adhesion on TiNb substrates.. The *a*-BC:H film with the low boron content (B/C ratio = 0.03) exhibited the least platelet adhesion and activation because few platelets were present. In this study, films with high-hydrophilicity surfaces exhibited better blood compatibility. Overall results in this chapter showed that 0.03 is the optimum B/C ratio for an anti-thrombogenic surface. The synthesized *a*-BC:H films appear to be promising for the surface modification of blood-contacting devices.

In chapter 6, the summary of conclusion for each chapters and this dissertation was included. The future works for this dissertation are also stated.