Observations on the Failure Behavior in a Diamond-Like Carbon Coated Nitinol Vascular Stent under Cyclic Loading

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Abstract. We investigated a failure behavior of diamond-like carbon (DLC) coated metallic vascular stent under cyclic loading. We employed cyclic loading of compression and tension on a V-shaped stent unit coated with DLC thin film using a screw-driven tensile tester. It was observed that channel cracking and delamination of DLC film occurred intensively in the regions subject to the tensile strain during cyclic loading. We investigated the pre-cleaning effect on the interfacial delamination behavior by the cross-sectional analysis of the fractured region using a dual beam FIB/SEM system.

Introduction

A stent is a small, lattice-shaped, metallic tube inserted into an abnormally narrowed or closed conduit (such as an artery or duct) in the body. The vascular stent helps hold an artery opened by its circumferential expansion to a large diameter so that blood could flow through it. The ideal stent requires not only mechanical properties of a sufficient radial strength and low recoil, but also a good biocompatibility of the stent surface for a lower thrombogenicity and restenosis [1]. In order to increase both mechanical properties and biocompatibility, a metallic stent would be coated with carbon [2], silicon carbide [3], titanium nitride [4], or tantalum [5]. During the expansion and contraction process of a film coated metallic stent, the stent may locally undergo a large tensile or compressive deformation causing critical failures in the coating layers, such as film cracking and interface delamination due to a misfit strain between a film and a metallic substrate [6, 7].

In the work, we investigated a failure behavior of Diamond-Like Carbon (DLC) coated on a metallic vascular stent under cyclic loading of compression and tension (see Fig. 1). A self-expandable vascular stent, made of nitinol (Ni-Ti alloy), were coated with DLC film, a protective coating due to its biocompatibility and excellent mechanical performance of high wear resistance and hardness. Cohesive channel cracking and delamination of DLC film were observed using Focused Ion Beam (FIB) system. We varied the adhesion strength with various voltage of Ar plasma.

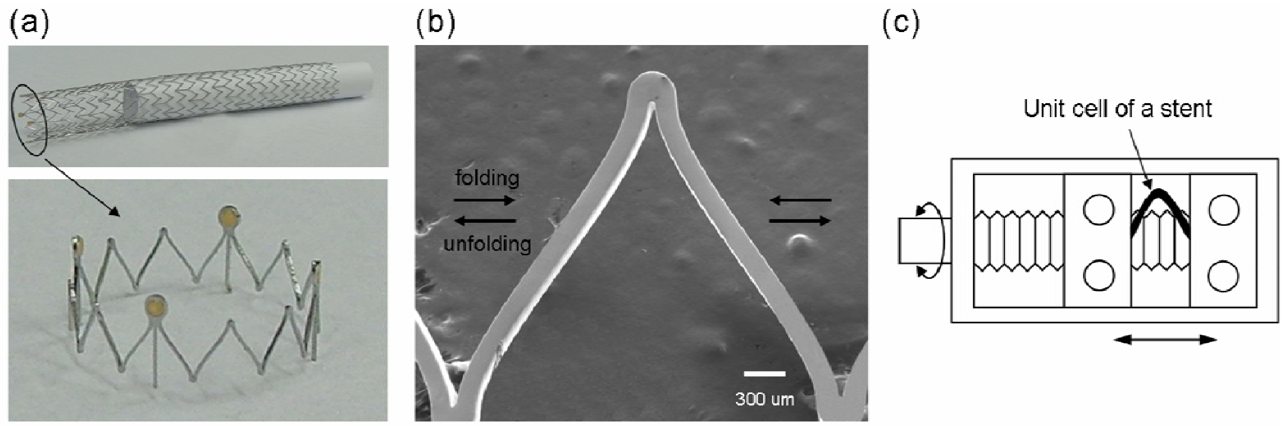

FIG. 1. (a) A self-expandable vascular stent (Length: 80 mm, Diameter: 10 mm), (b) a unit cell of a DLC coated self-expandable stent, (c) a schematic diagram of the unit cell of a stent holded on a screw driven tensile tester, applying cyclic tensile and compressive loading (the size of a stent unit is exaggerated).

Experimental

Specimen used in this work was provided from a self-expandable vascular stent (Zilver® 635™ Vascular Stent, COOK®). As shown in Fig. 1(a), the vascular nitinol stent was cut into a unit cell, of which the configuration of cross-section is rectangular shape with round corner edges. The unit cells of the stent were cleaned with acetone and ethyl-alcohol in ultrasonic bath. DLC films were deposited onto a unit cell of the stent by a radio-frequency plasma-assisted chemical vapor deposition (r.f. PACVD) with benzene (C_6H_6) gas (Fig. 1(b)).

In order to investigate failure behaviors with a function of the interface adhesion strength, a set of specimens were prepared for different deposition conditions of Ar plasma treatment from -200 to -800 V, followed by the deposition of Si interlayer at fixed condition of -200 V and 5 minute, which improves the adhesion strength between stent and DLC film. DLC film was deposited on Si interlayer at - 400 V and working pressure of 1.33Pa, resulting in the thickness and residual compressive stress of DLC film as 860- 940 nm and 0.8- 0.9 GPa, respectively.

The expansion and contraction process of a DLC coated nitinol stent during operation inside a vascular system were simulated with cyclic loading of tension and compression on a V-shaped unit cell using a screw-driven tensile tester. One cycle loading is carried out with a same amount of tensile and compression of 50%. At each step of cyclic loading, we explored the surface of DLC coated stent for tracing the nucleation and growth of cohesive cracking and interface delamination in DLC film using Dual Beam FIB/SEM system (Nova 200, FEI Company) which allows sectional analysis for the cross-section of delaminated region. For preventing the ion beam damage on the interest region with cracking or delamination, Pt layer was deposited prior to sectioning the delaminated region as shown in Fig. 3(c).

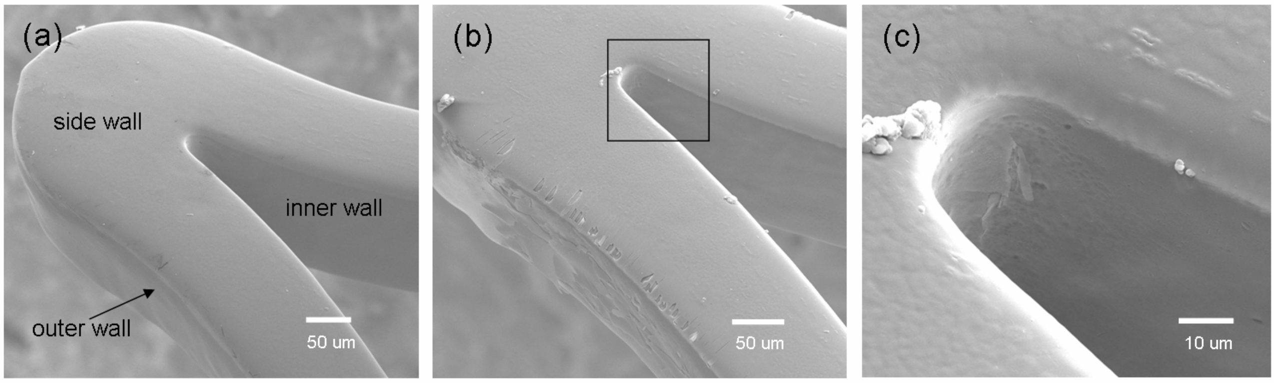

FIG. 2. DLC film with Ar plasma pre-cleaned at – 200 V (a) SEM image of a DLC coated stent before loading. (b) SEM image for intensively cracked region after one cyclic compressive and tensile loading. (c) Magnified region of square box in (b): a valley in the inner wall.

Results and Discussion

Fig. 2 shows the DLC coated vascular stent before and after one cyclic loading. When a unit cell of the stent unit is strained, the stent transfer a force into the DLC film by a well-known shear lag mechanism [8-10]. The tensile stresses in film increase with straining the stent and reach the critical fracture strength of the film, so that cracking in DLC film appears. After one cyclic loading, film cracking and delamination of DLC film intensively occurred at outer wall of a V-shaped unit cell, while the film cracking were less observed for inner and side wall in Fig. 2 (b) and (c), implying that a major strain for the failure would be localized in outer wall and the valley in inner wall. From the stent geometry, on loading a stent, the outer wall and the valley region are placed under tensile strain and the inner wall as opposite side of the outer wall is under compressive strain. Thus, the tensile strain can be considered to exhibit the predominant factor responsible for the cracking failures, which is affected by a stent geometry and thickness [1].

Fig. 3 shows a failure behavior of a DLC film with various Ar plasma etching voltages in a same region of the stent after 15 times of a cyclic loading. In the outer wall, it did not show difference of crack density by Ar plasma etching effect since most of sample set were delaminated. However, the interface delamination and the spallation was shown to be significantly suppressed in the side wall region for the DLC coatings with Ar pre-treatment with -500 V and -800 V, implying that the interfacial adhesion strength improves for higher voltage of Ar pre-treatment.

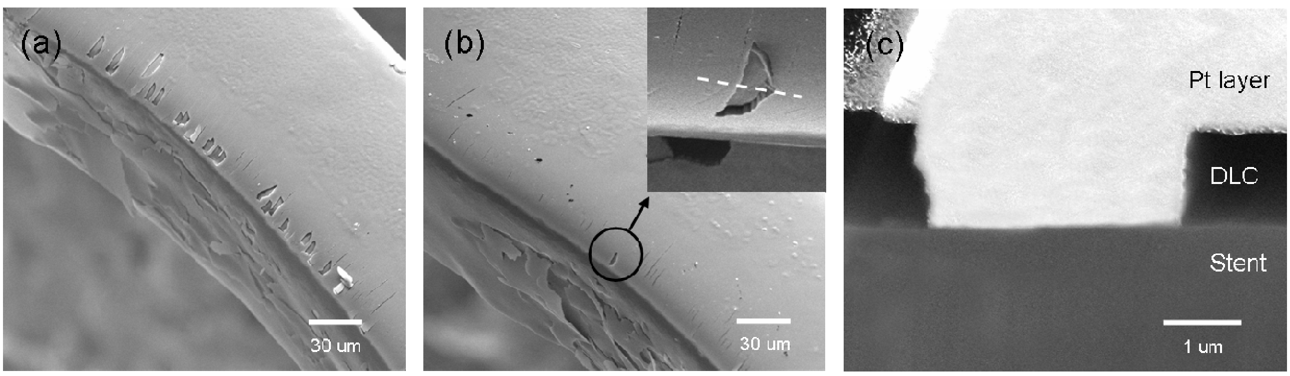

FIG. 3. SEM images of the outer side wall in a DLC coated stent after 15 cycle loadings. (a) Ar plasma etching at – 200 V, (b) at – 500 V, and (c) cross-section view on the delaminated region for an inset of (b).

The delaminated region, initiated from a cohesive crack (see the inset of Fig. 3(b)), were analyzed using FIB sectioning, revealing that DLC film was delaminated and spallated at the interface between film and stent substrate. The film spallation would be induced by the mixed mode (opening and shear mode) at interface as observed in a brittle film on relatively ductile substrate [11-13].

Summary

Failure behaviors of DLC film on a metallic vascular stent were investigated under the cyclic loading of repeated folding and unfolding on a V-shaped unit cell using a screw driven tensile tester and the coated surface was observed and analyzed using FIB/SEM system. Upon folding and unfolding a stent, a major strain for the failure is localized in outer wall and the valley in inner wall in which is intensively delaminated under a tensile strain. With higher voltage of Ar pre-treatment, the film cracking and spallation is significantly suppressed in the system. It was found that the delamination of film occurred at interface between film and stent substrate using FIB sectioning analysis.

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