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## Compositionally graded layer produced by low-energy ionization deposition technique

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#### **Abstract**

A carbon and silicon based thin layer was produced on a titanium alloy through the supply of accelerated molecular ions, which was called as an ionization deposition technique. Liquid materials of benzene ( $C_6H_6$ ) and hexamethyldisiloxane ( $C_6H_{18}Si_2O$ ) were individually evaporated under reduced pressure, and mixed in a chamber at a volume ratio changing with time. They were then ionized by an electron impact and accelerated in an electric field of 270 V m<sup>-1</sup> toward the alloy substrate. In this process, the ionized molecules were decomposed and fragmented to rearrange their bonding structure. X-ray photoelectron spectroscopic depth analysis revealed that the chemical bonding states of the layer changed from "graphite" to "silicon carbide + graphite" and "silicon carbide + graphite + titanium carbide", which corresponded to the decrease in C/Si atomic ratio from the surface side towards the substrate. From the results of Raman scattering analysis, both sp<sup>2</sup> and sp<sup>3</sup> hybrid bonding structures of carbons probably existed in the surface layer, which is similar to a hydrogenated amorphous carbon (a-C:H) structure. © 2005 Elsevier B.V. All rights reserved.

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#### 1. Introduction

Deposition techniques using ions and plasma, such as ion plating and ion beam deposition, which can realize low-temperature formation of hard and uniform surface modification layer, are practically applied to the hard coating to cutting

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tools, metallic molds, moving parts and so on. Coating onto not only metals or ceramics but also glasses or plastics is performed because of its low processing temperature. Materials that have been examined up to the present into details as the coating substance are limited to nitrides or carbides, such as TiN, TiC, CrN and SiC, and recently, diamond-like carbon (DLC) has become one of the next candidates as the advanced coating material [1–4]. Further, besides the monolayered coatings made of single-phase material as described above, multilayered coatings, e.g. TiN/AlN [5], composite coatings, e.g. Ti-Si-N, Ti-B-N [6,7], and metalincorporated DLC [8,9] have been investigated. In the coating process using solid-state sources, strict control of composition and chemical bonding state is difficult, and also the "droplet" phenomenon tends to happen, however, a report on the control of both the composition and the chemical bonding state along the depth direction of the layer has not been found so far.

In this research, a compositionally graded layer consisted of DLC-related carbon and silicon based material was produced by a low-energy ionization deposition technique, in which liquid-phase sources were evaporated, fragmented and partially ionized. This technique enables to control strictly

the composition and the chemical bonding state along the depth direction and gives smooth surface layer without the droplet. Depth-variation of the composition and bonding state, surface morphology and chemical structure were examined for the characterization of the compositionally graded layer obtained.

#### 2. Experimental

A substrate was a mirror-polished Ti-6Al-4V plate ( $Ra < 0.02 \,\mu\text{m}, 10 \,\text{mm} \times 10 \,\text{mm} \times 1.2 \,\text{mm}$ ). Ar ion bombardment was performed for 15 min in order to clean up the surface of the substrate. The ionization deposition apparatus is mainly comprised of a process chamber and a vacuum pumping system. The schematic diagram of the process chamber of the apparatus is shown in Fig. 1. The chamber equips an ionization area, a reactive-gas inlet and a substrate holder. Liquid sources of benzene (C<sub>6</sub>H<sub>6</sub>, >99.9% in purity) and hexamethyldisiloxane (abbreviated as HMDSO; C<sub>6</sub>H<sub>18</sub>Si<sub>2</sub>O, >99.0% in purity) were individually evaporated under reduced pressure and introduced to the chamber through the reactive-gas inlet at a volume ratio changing with time, as described in

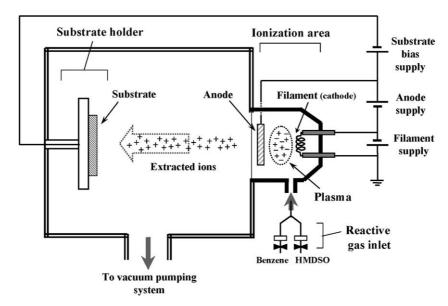


Fig. 1. Schematic diagram of the process chamber of the ionization deposition apparatus.

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