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（様式第５号）

アルミニウムバッファ層上に堆積した超ナノ微結晶ナノダイヤモンド膜の機械特性と化学結合構造との相関の解明
（英語名）
Study on relationship between mechanical properties and chemical bonding structures of ultrananocrystalline diamond films deposited on aluminum buffer layer

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※２　利用情報の公開が必要な課題は、本利用報告書とは別に利用年度終了後２年以内に研究成果公開｛論文（査読付）の発表又は研究センターの研究成果公報で公表｝が必要です（トライアル利用を除く）。
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１．概要（注：結論を含めて下さい）

Ultrananocrystalline diamond/amorphous carbon composite (UNCD/a-C) films were deposited by coaxial arc plasma deposition (CAPD) on cemented carbide (WC-Co) substrates directly and with Al interlayer. The films deposited on Al interlayer achieved evidently enhancement in hardness to 58 GPa and Young’s modulus to 613 GPa comparing with hardness of 50 GPa and Young’s modulus of 520 GPa of films deposited directly on the substrate. This significant improvement in the mechanical properties is consistent with enhanced C-C sp^3 fraction in the films. The Al interlayer successfully suppressed the Co catalytic effects that induces films graphitization and degrades films hardness and Young’s modulus.

２．背景と目的

Diamond and related hard carbon coatings have attracted attention because they are effectively applicable to cutting non-ferrous and abrasive materials due to their unique features such as high hardness and excellent mold release. The physical properties of carbon materials are strongly influenced by the ratio of sp^3 (diamond-like) to sp^2 (graphite-like) bonds. Ultrananocrystalline diamond/amorphous carbon composite (UNCD/a-C) films deposited on WC-Co at room substrate-temperature exhibit the maximum hardness of 50 GPa and a modulus of 520 GPa [1]. It was demonstrated that low substrate temperature is effective to diamond growth and suppressing the Co catalytic effects at the interface. Furthermore, the diffusion of Co atoms into the films hardly occurs even at the substrate temperature of 550 ºC. To further understand the role of Co catalytic effects on the properties of the films, Al interlayers were sputtered prior to the film deposition, because it was reported that the insertion of Al interlayers suppresses the catalytic reactivity of transition metals for inducing graphitization [2, 3]. The effects of Al interlayer on the films growth, the hardness, and Young’s modulus of the films were studied. In addition, the chemical bonding structures of the films were investigated by X-ray photoemission spectroscopy and near-edge X-ray absorption fine-structure spectroscopies to understand the physical origin of the films properties due to insertion of Al interlayer.

３．実験内容（試料、実験方法、解析方法の説明）

UNCD/a-C films were deposited on WC-Co substrates with dimensions of 10 mm diameter and 5.5 mm thickness at room substrate-temperature by CAPD with pure graphite targets. The inside of a vacuum apparatus was evacuated to base pressures of less than 10^{-4} Pa by a turbo molecular pump. An arc plasma gun equipped with a 720 μF capacitor was operated at an applied voltage of 100 V. The films were deposited directly and with Al interlayer sputtered at thickness of 100 nm. The hardness and Young's modulus of the deposited films were investigated by nano-indentation as shown in Fig.1. The deposited films were characterized by X-ray photoemission spectroscopic at beamline 12 of Kyushu Synchrotron.

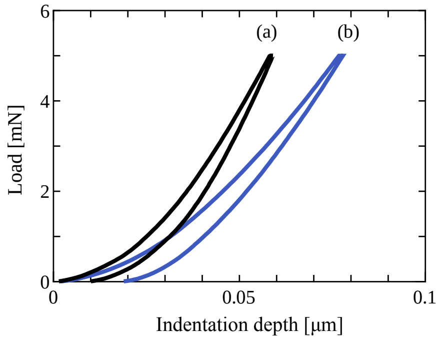

Fig. 1 Load–displacement curve of UNCD/a-C films deposited on WC-Co substrates (a) without and with Al interlayer.

４．実験結果と考察

The films deposited directly on WC-Co substrates exhibit a hardness of approximately 50 GPa and Young's modulus of 520 GPa. By the insertion of Al interlayers, the hardness and Young's modulus were improved to 58 GPa and 613 GPa, respectively. This improvement might be attributed to the suppression of Co catalytic effects that induces films graphitization and in turn reduces films hardness and Young's modulus.

Figure 2 shows the elemental composition of UNCD/a-C films measured by XPS survey. O 1s and C 1s peaks were detected, which indicates that the films are free of substrate contaminations. The O 1s peak is attributed to the residual oxygen in the chamber during the films preparation and exposure to the environmental air after deposition [4]. Figure 3 shows the X-ray photoemission spectra of UNCD/a-C films, which were decomposed to peaks by a software. The estimated $sp^3/(sp^3+sp^2)$ ratio show an enhancement due to the insertion of Al interlayer, which show an agreement with the measured hardness and Young's modulus. The Al interlayer suppresses the catalytic reactivity of Co that induces films graphitization.

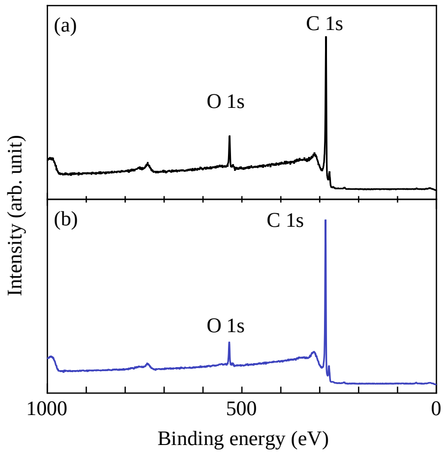

Fig. 2 XPS survey of UNCD/a-C films on WC-Co substrates (a) without and with Al interlayer.

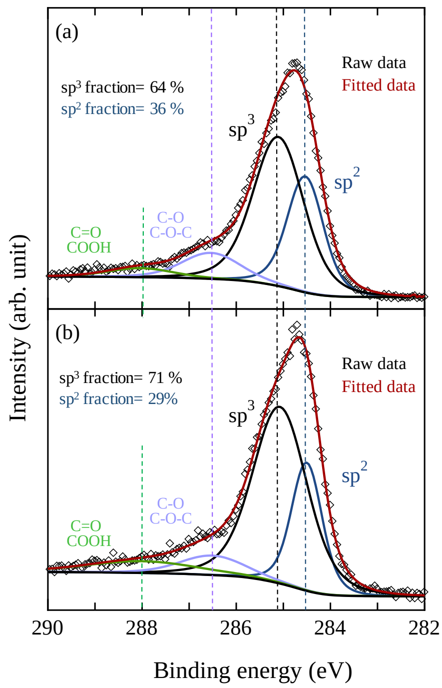

Fig. 3 C 1s X-ray photoemission spectra of UNCD/a-C films deposited (a) without and (b) with Al interlayer.

５．今後の課題

In this work, the role of Al interlayers in the suppression of Co catalytic effects, and the relationship between the films hardness and C-C sp^3 fraction were investigated. From this, we are going to study the effects of the Al interlayer insertion on the adhesion strength of the film with the substrates and the film surface morphologies.

６．参考文献

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７．論文発表・特許（注：本課題に関連するこれまでの代表的な成果）

- Hiroshi Naragino et al., Evergreen, 03, 1-5 (2016)
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８．キーワード（注：試料及び実験方法を特定する用語を２～３）

Nanodiamond, Coaxial arc plasma, CAPD, Hardness, XPS, X-ray photoemission spectroscopy, Al interlayer

９．研究成果公開について

①　論文（査読付）発表の報告　　　　　（報告時期：**2020** 年 **3** 月）