

The Stress and XPS Studies of B₄C Coating by Reactive Sputtering

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In X-ray free-electron laser (XFEL), coating mirrors based on the principle of total external reflection are the important optical elements. It not only can realize the high reflectivity but also maintain the x-ray wavefront. Boron carbide (B₄C) is a superior material possessing low absorption coefficient, high hardness, and high damage threshold. However, B₄C coating has strong compressive stress. Reactive sputtering technique is a general method to reduce the coating stress. 50-nm-thickness B₄C coatings were deposited by reactive sputtering with various nitrogen-argon mix ratios. The nitrogen proportion was 4%, 8%, and 15%, respectively. The interferometer measurement results showed that when the nitrogen proportion was increased from 0% to 15%, the stress of B₄C coating was decreased from -3.06 GPa to -0.84 GPa. It was explained by the chemical environment and the bonding structure of boron atoms. The X-ray photoelectron spectroscopy results showed that as compared with pure B₄C coating, boron atoms of nitride B₄C coating were mainly existed in the form of B-N and B-C bonds, corresponding to BN and B₄C compounds, respectively. When the nitrogen proportion was 15%, the B-N bond content was increased to 79% and had been higher than the B-C bond content. It demonstrated that the introduction of nitrogen atoms inhibited the formation of B-C bonds. Meanwhile, the atomic ratio of boron to carbon was decreased to about 3.3:1 in B₄C coating. This trend was consistent with the fitting density of B₄C coatings measured by grazing incidence X-ray reflectometry (GIXRR). In GIXRR measurement, the B₄C coating prepared under 8% of nitrogen proportion had 93% reflectivity at 0.154 nm wavelength, which was of interest for application in X-ray free-electron laser.