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Magnetron sputtered Hf–B–Si–C–N films with high oxidation resistance in air above 1500 °C

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The present work focuses on the effect of nitrogen addition into Hf–B–Si–C films in order to significantly improve the oxidation resistance of these films. Hf–B–Si–C–N films were deposited onto Si(100) and SiC substrates using pulsed magnetron co–sputtering of a single B_4C–Hf–Si target (at a fixed 15% Hf fraction and a 20% Si fraction in the target erosion area) in an Ar+N_2 gas mixture at the N_2 fraction ranging from 0% to 50%. A planar unbalanced magnetron was driven by a pulsed dc power supply operating at a repetition frequency of 10 kHz with an average target power of 500 W in a period and a fixed 85% or 50% duty cycle. Substrates were held at a floating potential and a temperature of 450 °C. The effect of the N_2 fraction in the gas mixture and of the duty cycle on structure and properties of the films was investigated. The increasing N content (from 0 to 52 at.%) in the films was compensated by decreasing contents of B (from 39 to 24 at.%), Si (from 24 to 15 at.%), Hf (from 25 to 4 at.%) and C (from 7 to 3 at.%). The structure of the Hf–B–Si–C film prepared in pure argon was nanocomposite and admixture of N_2 into the gas mixture resulted in X-ray amorphous Hf–B–Si–C–N films. All films exhibited high hardness in the range of 17–21 GPa. Increase in the N_2 fraction in the gas mixture led to a rapid rise in the electrical resistivity of the films up to non-measurable values for N_2 fractions >15%. Differences in structure and properties of Hf–B–Si–C–N films prepared at the 85% and 50% duty cycles were negligible. Based on the measurements of mechanical properties and electrical conductivity of the Hf–B–Si–C–N films investigated, the $Hf_7B_{25}Si_{21}C_5N_{40}$ film with 2 at.% of Ar possessing hardness of 20 GPa and electrical resistivity of 2×10^{-1} Ωm was selected for oxidation tests in synthetic air up to 1700 °C. The film exhibited very high oxidation resistance even above 1500 °C due to a nanocomposite protective layer on the surface consisting of HfO_2, HfB_2 and Hf(C,N) nanocrystallites surrounded by a SiO_2–based amorphous matrix, most probably containing boron as B_2O_3 (borosilicate glass).

Keywords
Hf–B–Si–C–N films
Pulsed magnetron sputtering
Oxidation resistance