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DSC Analysis of Nanocomposite ZrO₂-Al₂O₃ Coatings

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The ZrO₂-Al₂O₃ coatings were deposited at various coating compositions, ranging from pure zirconia to 50% alumina content, by a reactive pulsed-DC magnetron sputtering technique using two 50 mm diameter Zr and Al targets. The coating with a Zr-Al-O solid solution structure was composed of cubic-ZrO₂ and alumina distributed in the nano-size ZrO₂ grains (10-20 nm depending on the aluminum content). The deposited coatings were studied using high temperature Differential Scanning Calorimetry (DSC) up to 1350°C, X-ray diffraction (XRD) and High Resolution Transmission Electron Microscopy (HRTEM). It was found that after annealing, two exothermic events were observed. The first event appeared at 600°C and was completed at 800°C leading to alumina segregation from the solid solution phase into the grain boundaries, and the formation of the nc-ZrO₂/a-Al₂O₃ structure; while the second event appearing at 1000-1200°C was related to grain growth and transformation of the coating from a stabilized cubic to a monoclinic phase. The coating hardness after the first event was stabilized to 19.5±1.1 GPa value because of the segregation effect, which hindered the grain growth. However, after a second DSC cycle of the coating only one sharp endothermic transformation peak was observed at 1100°C which was attributed to zirconia transformation from monoclinic to tetragonal phase. The results show that in the case of zirconia-alumina coatings, the formation of the nc-ZrO₂/a-Al₂O₃ structure (nc-nanocomposite, a-amorphous) is governed by the driving force of the Al₂O₃ segregation because of the immiscibility of the two phases.

Keywords

magnetron sputtering

DSC

XRD

HRTEM