Morphological changes of the PVD coatings after isothermal annealing

Paulína Babincová, Martin Sahul, Peter Drobný, and Ľubomír Čaplovič

Slovak University of Technology, Faculty of Materials Science and Technology, Jána Bottu 25, Trnava, Slovakia

The main of this work is to investigate the impact of the isothermal annealing on the structure and morphological changes on the PVD hard coatings. Three different coatings based on the AlTiN system were deposited onto cemented carbide substrates containing 6 wt% of Co. Two coatings AlTiN/TiAlN and AlTiN/TiN with an initial thickness of 3 μm had a coarse-grained columnar structure with a chemically graded nanomultilayering consisting of alternating Ti-rich and Al-rich layers. In the case of the AlTiN/TiAlN coating, a total of 20 layers were applied. 10 layers had a thickness of 200nm (AlTiN) and 10 layers with a thickness of 100 nm (TiAlN). The same number of nanolayers was also retained for the AlTiN/TiN multilayer coating. The TiN layers were 100 nm thick. The third coating was a nanocomposite sandwich coating formed by a TiSiN adhesive layer (50nm), a functional nanocomposite AlTiSiN layer (2 μm) and a top functional TiSiN layer with a thickness of 1 μm. The coatings were prepared by cathodic arc evaporation using the LARC$^{\®}$technology. After deposition, the samples were annealed at high temperatures (700°C, 800°C, 900°C, 1000°C) for one hour in air. With X-ray diffraction and scanning electron microscopy, the morphology, structure and phase composition of coatings and oxide layers after annealing were evaluated.

After the heat-treatment at 800°C, cross-sectional and surface morphology images revealed, that an oxide layer has grown on top of all coatings. EDS analysis showed that the oxide layers on the multilayers coatings had a layered structure, which was found to be the consequence of the high diffusion rate of Al. The interface with the coatings was very rough. It was depleted of Al, which diffused to the upper surface of the oxide. The oxidation process of nanocomposite AlTiSiN/TiSiN coating started by forming an oxide layer of ternary Ti-Si-O at the interface. This system segregated into two phases of TiO_2 and SiO_2. The nanocomposite coating remained stable even after annealing at 1000 ° C. The surface of the multilayers was accompanied by a network of cracks at this temperature. An interesting phenomenon was also observed on the substrates itself during the annealing. As a result of oxidation, unprotected sides of the substrates expanded. Globular oxides based on tungsten and cobalt were also formed on the surface of the samples at the places where the coating was peeled off. The reasons for the differences are discussed.

This work was supported by the Project of VEGA Grant Agency of the Ministry of Education, Science, Research and Sport of the Slovak Republic and Slovak Academy of Sciences, No. 1/0540/19: "Research of possibilities of increasing the thermal and oxidation stability of Al-Ti-N based hard coatings" and also by the Slovak Research and Development Agency with grant number APVV-15-0168: "Research of modification of phase interfaces in the coating/substrate system to increase the adhesion of hard coatings".