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Thermal stability of TiZrAlN films deposited by a reactive magnetron sputtering method

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The aim of the present work is to study the thermal stability, under vacuum and air annealing, of quaternary transition metal nitride films, namely TiZrAlN, with emphasis on the role of Al content on the structure and phase formation. $(Ti,Zr)_{1-x}Al_xN$ films with thickness of 300 nm have been deposited onto Si (001) wafers by a reactive unbalanced magnetron sputtering method. Ti, Zr and Al targets were co-sputtered under mixed $Ar+N_2$ plasma discharges. Varying the RF power of the Al target from 20 to 200 W resulted in a concentration of Al (x_{Al}) in the films to increase from 2.6 to 36.4 at.%, while the Ti:Zr concentration ratio was kept constant to ~1.0. Results of the XRD analysis on as-deposited films indicate the formation of (Ti,Zr,Al)N solid solution. However, the structure changes with rising Al concentration from crystalline to nanocrystalline and then it turns into the amorphous state. Annealing in vacuum (~10^{-4} Pa) at the temperature of 600°C doesn't cause any essential change of (Ti,Zr,Al)N solid solution structure. After annealing in vacuum at 950°C, the structure of the films depends essentially on the aluminum content. At the smaller Al concentrations, the (Ti,Zr,Al)N solid solution remains stable. When x_{Al} > 9 ат.% the volume content of ZrN mononitride phase increases and when x_{Al} > 13 ат.% the TiN and ZrN phases become dominating. At the same time, Al is apparently included substitutionally into these phases by forming the solid solutions of TiAlN and ZrAlN type. Annealing under air atmosphere of the investigated TiZrAlN samples was carried out for temperature intervals ranging from 400 to 950°C using in situ temperature XRD. Decomposition of the (Ti,Zr,Al)N solid solution already starts at the temperature of 500°C and the diffraction lines corresponding to a solid solution disappear at T = 600°C. The intensive formation of ZrO_2, TiO_2 and Al_2O_3 oxide phases occurs at a temperature of 780°C. Increase in x_{Al} up to 25 at.% leads to oxide formation reduction that can be explained by the passivating role of the Al_2O_3 surface layer. Thus, higher oxidation resistance of TiZrAlN films is reached in a case of high Al concentration at which amorphization of their structure takes place.

Keywords
nanocomposite TiZrAlN
thermal stability
solid solution