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Structural evolution in reactive RF magnetron sputtered (Cr,Zr)2O3 upon annealing

Ludvig Landälv[1], Jun Lu[2], Steffi Spitz[3], Harald Leiste[3], Sven Ulrich[3], Mats Ahlgren[4], Emmanuelle Göthelid[4], Lars Hultman[2], Michael Stueber[3], Per Eklund[2]

[1]Linköping university / Sandvik Coromant, Linköping, Sweden [2]Linköping University (LiU), Linköping, Sweden [3]Karlsruhe Institute of Technology (KIT), Karlsruhe, Germany [4]AB Sandvik Coromant, Stockholm, Sweden

ludla@ifm.liu.se

Physical vapor deposited binary oxide alloys has drawn attention in the past years, often focusing on the Al-Cr-O system [1,2]. The interest for this material system stems from the possibility to stabilize the desired corundum phase, α-Al_2O_3, by introducing other elements to the alloy such as Cr in α-$(Al,Cr)_2O_3$.The α-structure is stabilized by means of a template growth; Cr forms escolaite Cr_2O_3 which is isostructural with corundum. Exchanging Al with Zr, which is used in many other ceramic alloy systems, creates a new and interesting oxide system with the retained stabilization from Cr, despite Zr's one higher valence than Al and being significantly larger in size. Spitz et al. mapped the Cr-Zr-O system over a wide range of Cr/Zr composition by reactive RF-magnetron sputtering [3], and showed how the system exhibits different phases: solid solution (SS) in corundum structure at low Zr-content, cubic-$(Zr,Cr)O_2$ based SS at ~50 at % Zr, and monoclinic/tetragonal SS $(Zr,Cr)O_2$ for higher Zr-content.
In the present study, high Cr-containing $(Cr,Zr)_2O_3$ thin films were synthesized in a corundum structure, at 500 °C, to a thickness of about 4.5 µm. The films were then vacuum annealed up to 810 °C for 5 h. Characterization of the hardness, phase and microstructure were performed using a combination of Vickers hardness, X-ray diffractometry and TEM + EDX on the atomic-to-nm scale.
We observe phase transformations with the initial formation of a banded microstructure of Cr-rich and Zr-rich oxide lamellas, containing small crystallites. This nanostructure eventually coarsens. Correlated with the nucleation and growth of tetragonal-ZrO_2 phase is an age-hardening behavior. Annealing the sample to 750°C thus results in a hardness increase from 600 Hv05 for the as-deposited sample (500°C) to 1900 Hv05. Films annealed to 810°C exhibits a hardness of 980 Hv05.
[1] Ramm, J., et al., Surf. & Coat. Tech., 2007. 202(4–7), [2] Khatibi, A., et al. Acta Mat., 2013. 61(13) [3] Spitz S., et al. Thin Solid Films, 2013, 548

Keywords
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