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In-Situ²: In-Situ High-Temperature High-Energy X-Ray Diffraction and In-Situ Micromechanical Testing of nanostructured AlCrSiN-AlCrN multilayer coating prepared by reactive arc evaporation

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Nanostructured hard coatings exhibit complicated structure-property relationships which originate from their microstructure, phase and residual stress variations at the nanoscale. In this contribution, a multi-layered coating composed of equally thick soft hexagonal (h) Al₉₀Cr₁₀N and metastable hard cubic (c) Al₆₃Cr₂₇Si₁₀N sublayers, with a bilayer period of ˜65 nm, was investigated during annealing by high-temperature high-energy synchrotron X-ray diffraction (HT-HE-XRD) and complementary micromechanical tests in a scanning electron microscope (SEM). HT-HE-XRD was performed by using an in-situ dilatometry setup, which has, due to size restrictions, not been established up to now in the field of hard protective coatings. A combination of high-temperature dilatometry and high-energy small- and wide-angle synchrotron X-ray diffraction (SAXD, WAXD) represents a unique tool for characterization of coatings, revealing microstructure development at high temperatures accompanied by variations of residual stresses. Both types of data analysis evidenced that the periodicity of the multi-layered structure changes with the onset of the cubic-to-hexagonal transition in the Al₆₃Cr₂₇Si₁₀N sublayers. SEM on the coating cross-section before and after the thermal cycle indicates that the multilayer architecture remains stable, whereas the bilayer period increases to ˜73 nm. Furthermore, in situ micromechanical tests on cantilevers fabricated using focused ion beam (FIB) milling from the as-deposited and annealed state were used to determine Young's modulus *E*, fracture stress *σ*_F and fracture toughness *K*_{Ic}. The measured mechanical properties proved, that during annealing the elastic properties (*E*) change with phase transformation, whereas the fracture properties (*σ*_F, *K*_{Ic}) remain nearly constant after the thermal cycle. This evidences that the fracture properties depend mainly on the microstructure, not as much on the elastic difference between the constituents of the system.

Primary author: Mr MEINDLHUMER, Michael (Christian Doppler Laboratory for Advanced Synthesis of Novel Multifunctional Coatings at Department of Physical Metallurgy and Materials Testing, Montanuniversität Leoben)

Co-authors: Mr KLIMA, Stefan (Christian Doppler Laboratory for Advanced Synthesis of Novel Multifunctional Coatings, Department of Physical Metallurgy and Materials Testing at Montanuniversität Leoben); JÄGER, Nikolaus (Christian Doppler Laboratory for Advanced Synthesis of Novel Multifunctional Coatings at Department of Physical Metallurgy and Materials Testing, Montanuniversität Leoben); Dr STARK, Andreas (2Helmholtz Zentrum Geesthacht, Centre for Materials and Coastal Research, Geesthacht,); Dr HRUBY, Hynek (eifeler-Vacotec GmbH); Prof. MITTERER, Christian (Christian Doppler Laboratory for Advanced Synthesis of Novel Multifunctional Coatings at the Department of Physical Metallurgy and Materials Testing, Montanuniversität Leoben); Prof. KECKES, Jozef (Department of Material Physics, Montanuniversität Leoben); Prof. DANIEL, Rostislav (Christian Doppler Laboratory for Advanced Synthesis of Novel Multifunctional Coatings at the Department of Physical Metallurgy and Materials Testing, Montanuniversität Leoben)

Presenter: Mr MEINDLHUMER, Michael (Christian Doppler Laboratory for Advanced Synthesis of Novel Multifunctional Coatings at Department of Physical Metallurgy and Materials Testing, Montanuniversität Leoben)

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