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Visualising microstructural dynamics of titanium aluminium nitride coatings under variable-temperature oxidation

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Background incl. aims

Titanium-aluminium nitride (Ti1-xAlxN) coatings are used to achieve high oxidation and wear resilience in coated metal cutting tools. For the highest performance, the coatings' composition and microstructure are carefully controlled. However, it is difficult to obtain knowledge how these factors effect of the oxidation processes at the microstructural level because studies are most commonly performed: (1) post mortem and cannot observe the structural dynamics underlying the oxide formation in real-time; or (2) averaged over a large volume of the sample (and use powdered materials rather than actual coatings) and lack the spatial resolution to form connections to microstructure. Here, we describe the use of ETEM for visualizing the oxidation process at high spatial and temporal resolution, using technically relevant materials, to provide important insight into where oxidation initiates and how it proceeds to determine the performance of the coating.[1]

Methods

Industrial style Ti1-xAlxN coatings were grown on WC-Co substrates with x = 0, 0.18, 0.44, & 0.67. Samples were prepared through focused ion beam milling (fig. 1a), transferred to MEMS heating chips (Norcada Inc., fig 1b), and finally imaged in ETEM (Hitachi HF3300-S, operated at 300 kV) during heating in an O2 environment (ca. 1 Pa). The samples were observed over a temperature range from 200 to 1000 °C and energy dispersive X-ray spectroscopy (EDX) with pre- and post- elemental mapping by EDX.

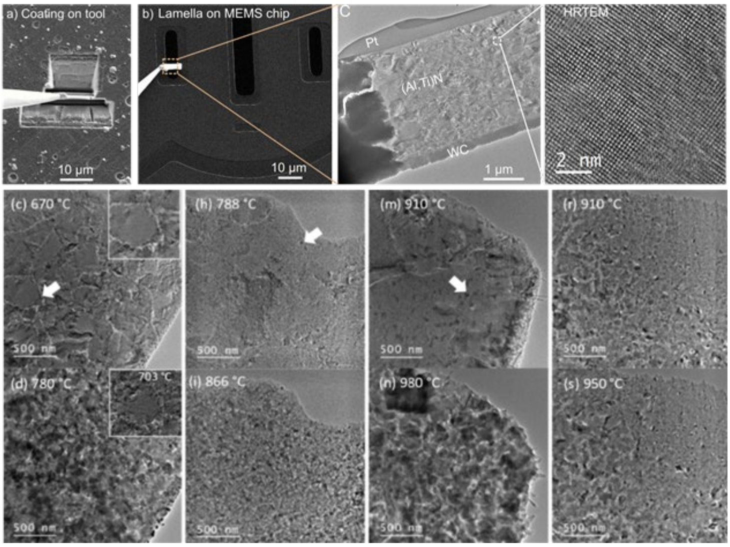
Results

The high-frame-rate ETEM movies show that oxidation in TiN proceeds at the grain boundaries and cracks formed during the heating process; in contrast, Ti1-xAlxN coatings transform from large as-deposited grains into oxide nanoparticles. The onset temperature increase with increasing Al content up to x= 0.44, as shown in fig. 1c-s. Moreover, high-resolution ETEM imaging show the presence of anatase TiO2 at the early stages of oxidation across all compositions. Above ~850 °C, the oxide nanoparticles grow through crystal merging, diffusion and recrystallization to form rutile TiO2. The EDX elemental maps coupled with secondary electron imaging reveal a uniform TiO2 sublayer decorated with increasing coverage of Al2O3 particles for x = 0.18 to 0.44. In contrast, coatings with x = 0.67 reveal a complete in-plane phase separation of Al- and Ti-oxides.[1]

Conclusion

The trend with increasing onset temperature for the oxidation with increasing Al content is consistent with the resilience of the corresponding coatings during cutting operations. The in-plane separation observed for the highest Al content (x = 0.67) exposes the softer TiO2 at the surface, and the TiO2-Al2O3 grain boundaries can act as a conduit for oxygen to reach further into the coating; together, these results can rationalise the poorer long-term oxidation resilience resulting from excessive Al.[1] Finally, the study provides insight into the real-time structural dynamics underpinning the oxidation resistance of Ti1-xAlxN coatings, illustrating how ETEM can uniquely complement other in situ techniques that have recently been applied to these materials.[2,3]

Graphic:



Keywords:

ETEM; TiAlN; coatings; oxidation; tooling

Reference:

[1] O.A. Makgae, F. Lenrick, V. Bushlya, J.M. Andersson, R. M'Saoubi, M. Ek, Appl. Surf. Sci. 618 (2023) 156625. https://doi.org/10.1016/j.apsusc.2023.156625.
[2] C. Saringer et al., Suf. Coatings Technol. 374 (2019) 617
[3] A.B.B. Chaar et al., J. Alloys Compd. 854 (2021) 157205