Karlsruher Institut für Technologie

IAM Institute for Applied Materials

Columnar grain boundaries are the weakest link in hard coatings

Insights from micro cantilever testing with bridge notches

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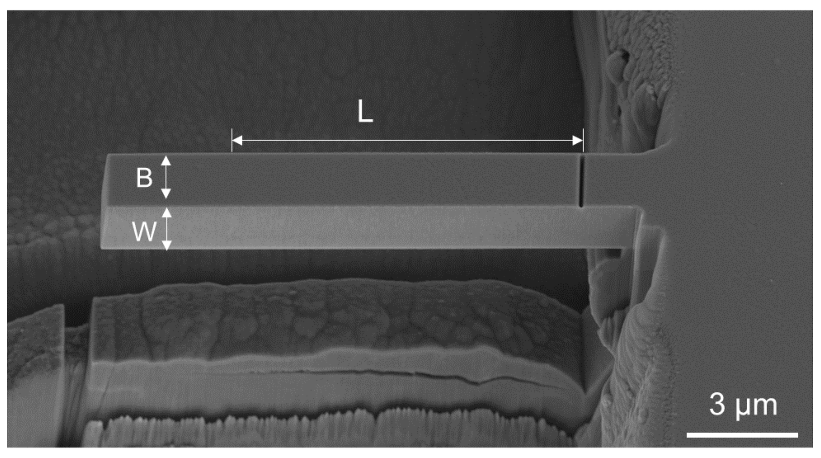
Motivation Hard coatings, particularly those prepared using physical vapor deposition (PVD) processes, are characterized by a columnar grain structure, resulting in elasticity heterogeneity and a high density of grain boundaries (GBs).There have been efforts to understand the effects of columnar GBs on the mechanical properties of hard coatings, and even to improve them via GB engineering. However, quantitative investigations of the effect of columnar GBs on the fracture toughness are still scarce.

Objective Provide a clear and fair comparison between specimen with and without GBs and to gain quantitative insights into the effect of columnar boundaries on the fracture toughness.

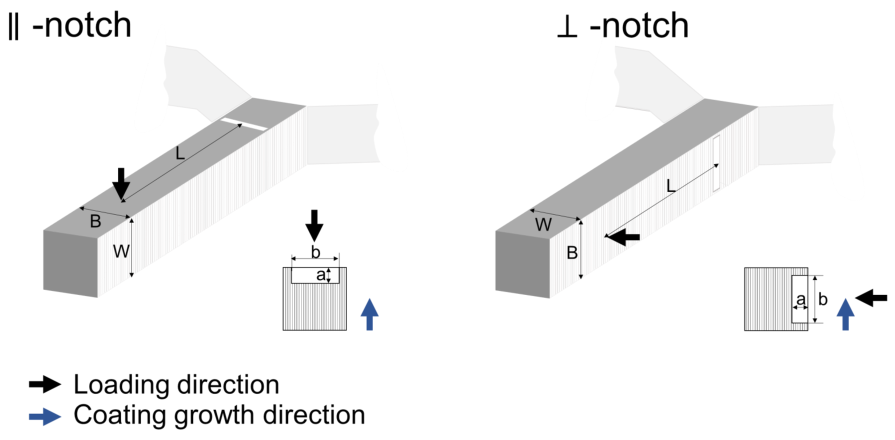
Methodology

Sample preparation

Thin film synthesis: the 4/2 nm CrN/AlN, CrN, AlN coatings are synthesized on Si (100) and MgO (100) substrate. Cantilevers are prepared with a focused ion beam, 3 nA and 700 pA at 30kV for coarse milling and 50 pA for fine milling. Pre-notches were fabricated with 20 pA current at parallel (∥-notch) and perpendicular direction (⊥-notch). The dimensions of the cantilevers were kept consistent with an *L*:*W*:*B* ratio of 5:1:1.



The free-standing cantilever of the cg/epi-CrN/AlN



Schematic illustration of a microcantilever geometries with different loading directions

Mechanical testing

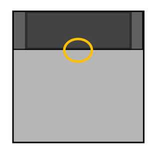
Determine the toughness at parallel and perpendicular direction with an *in situ* SEM indenter bending fracture tests. A Hysitron PI 89 PicoIndenter equipped with a 10 µm wide diamond wedge was used in displacement controlled mode, at 5 nm/s.

Data analysis

- The fracture toughness at point C, K_{IC}, was calculated using Matoy's model,

(Matoy *et al.*, Thin Solid Films, 2009)

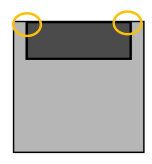
$$K_{IC} = \frac{F_C \cdot L}{B \cdot W^{3/2}} \cdot f_{Matoy}\left(\frac{a}{W}\right)$$

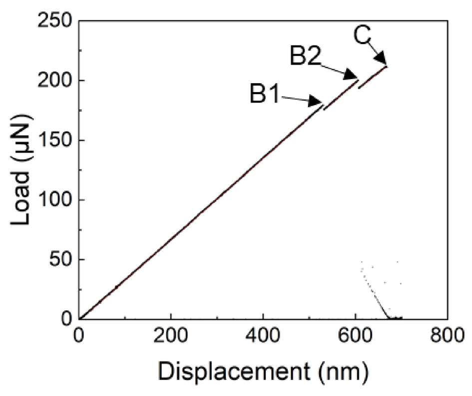


- At point B1 and B2, the fracture toughness at bridges, K_{IC}^*, was calculated using Brinckmann´s correction,

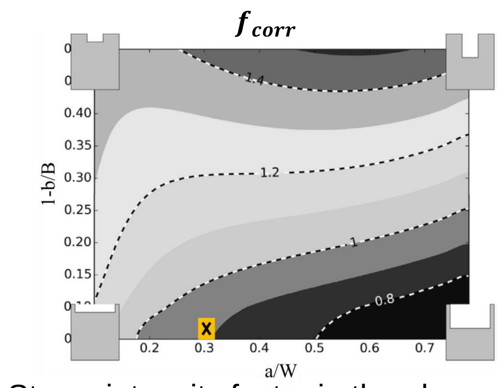
(Y. Zhang, et al., Mater. Des. 2023)
(Brinckmann *et al.*, Acta Mat. 2017)

$$K_{IC}^* = \frac{F_B \cdot L}{B \cdot W^{3/2}} \cdot \frac{1}{f_{corr}} f_{Matoy}\left(\frac{a}{W}\right)$$





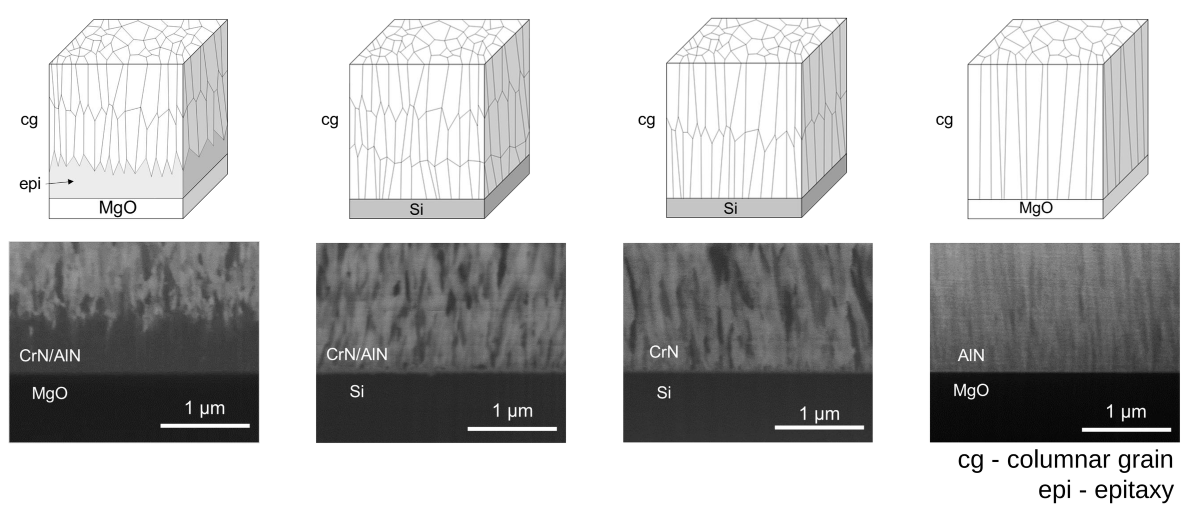
Load-displacement curves from a single test on cg-CrN/AlN coatings.



Stress intensity factor in the absence of a bridge divided by the average stress intensity top half of the bridge.

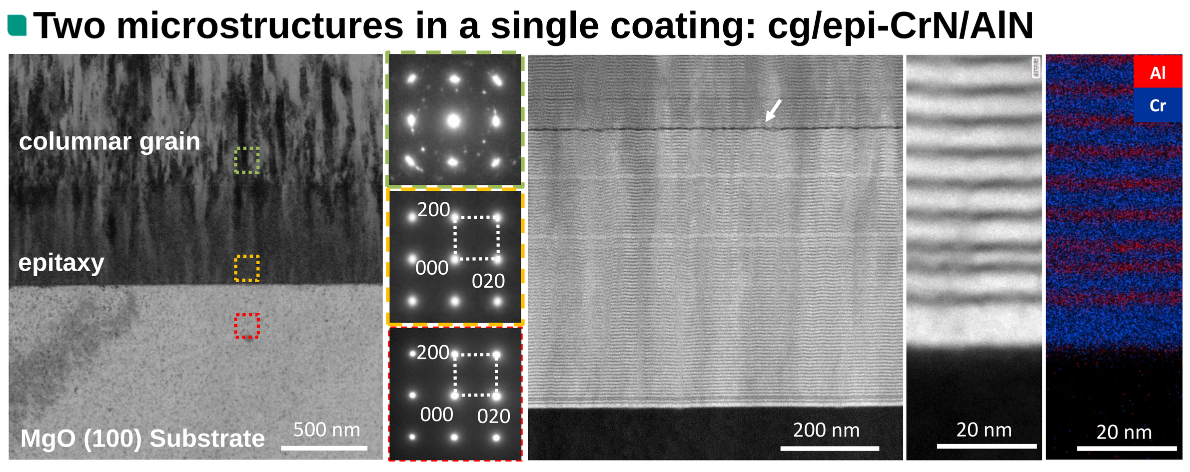
Microstructure investigation

- cg/epi-CrN/AlN
- cg-CrN/AlN
- cg-CrN
- cg-AlN



cg - columnar grain
epi - epitaxy

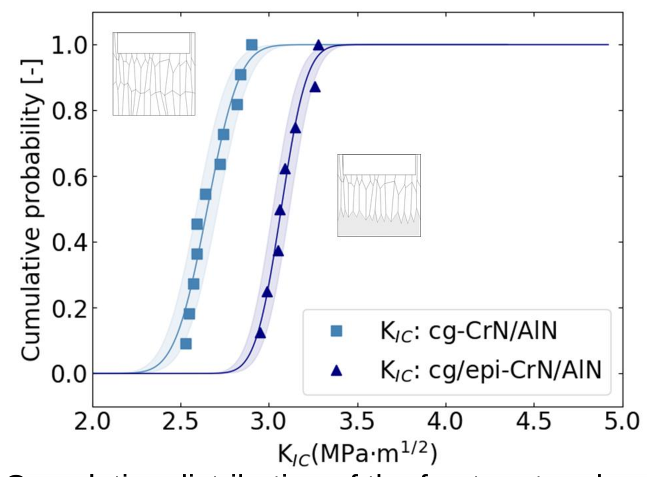
Two microstructures in a single coating: cg/epi-CrN/AlN



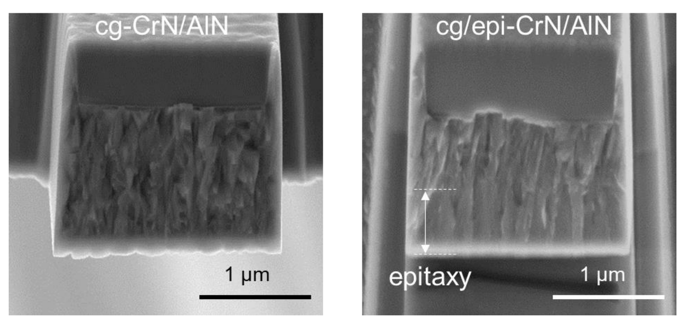
TEM by Ujjval Bansal at the Karlsruhe Nano Micro Facility (KNMFi)

Fracture toughness investigation

cg/epi-CrN/AlN vs cg-CrN/AlN

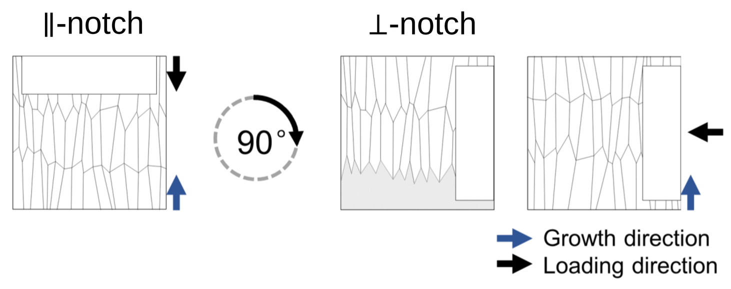


Cumulative distribution of the fracture toughness

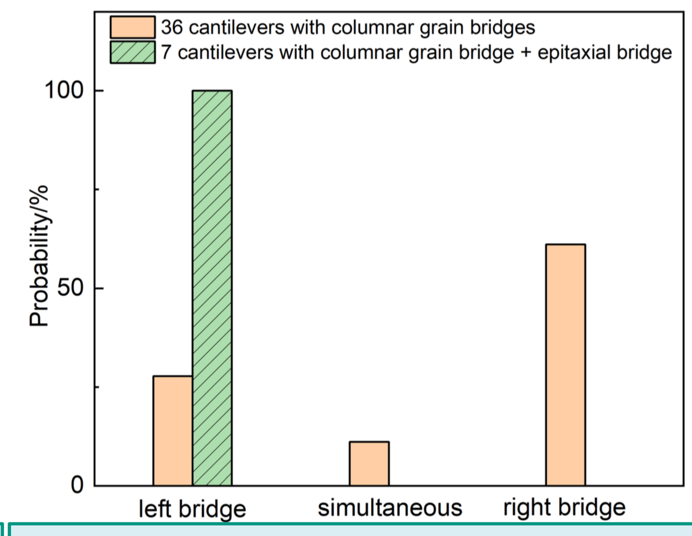


- Epitaxy contributed 15% increase.
- Qualitative comparison of epitaxy and columnar grain structure.

Schematics of the cantilevers

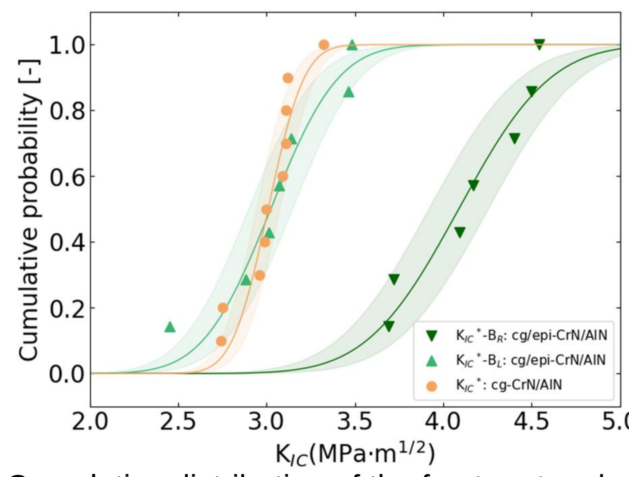


Bridge failure sequence

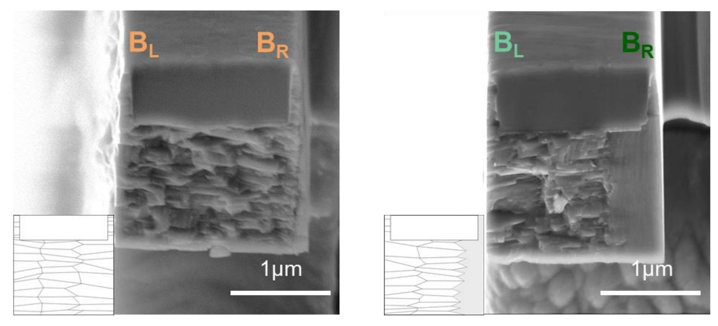


- The bridges at the columnar grain structure always fail first, which qualitatively indicates that columnar GBs are indeed the weakest link.

Local fracture toughness measurement



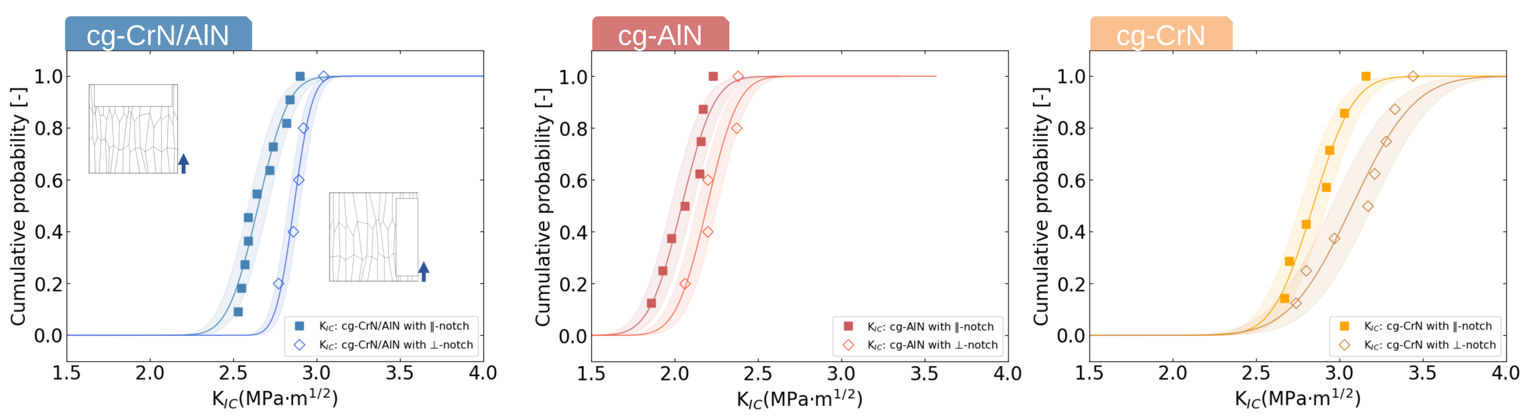
Cumulative distribution of the fracture toughness



- The epi-bridge has a higher fracture toughness of 4.1 ± 0.4 MPa·m$^{1/2}$ compared to the cg-bridge of 3.0 ± 0.3 MPa·m$^{1/2}$.

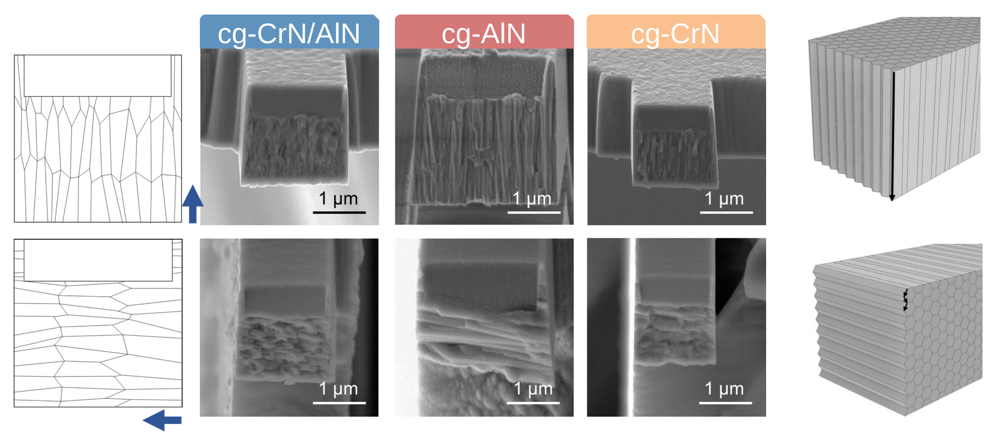
The effect of GB orientation

The fracture toughness at different loading direction



- Loading perpendicular to the growth direction results in 10% increase in K_{IC} compared to that loaded in parallel direction.
- The increase was observed in three different hard coatings.
- Despite the different loading direction, the area of the fracture surface is similar due to intergranular fracture and crack deflection. It suggests that the crack propagation direction plays a significant role.

The fracture surface



Conclusion and plan

- Grain boundary dominates fracture toughness and fracture behavior in PVD hard coatings
- Exploring the GBs effects on the hard coating at high temperature

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FWF Österreichischer Wissenschaftsfonds
Robert Bosch Stiftung
HELMHOLTZ

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